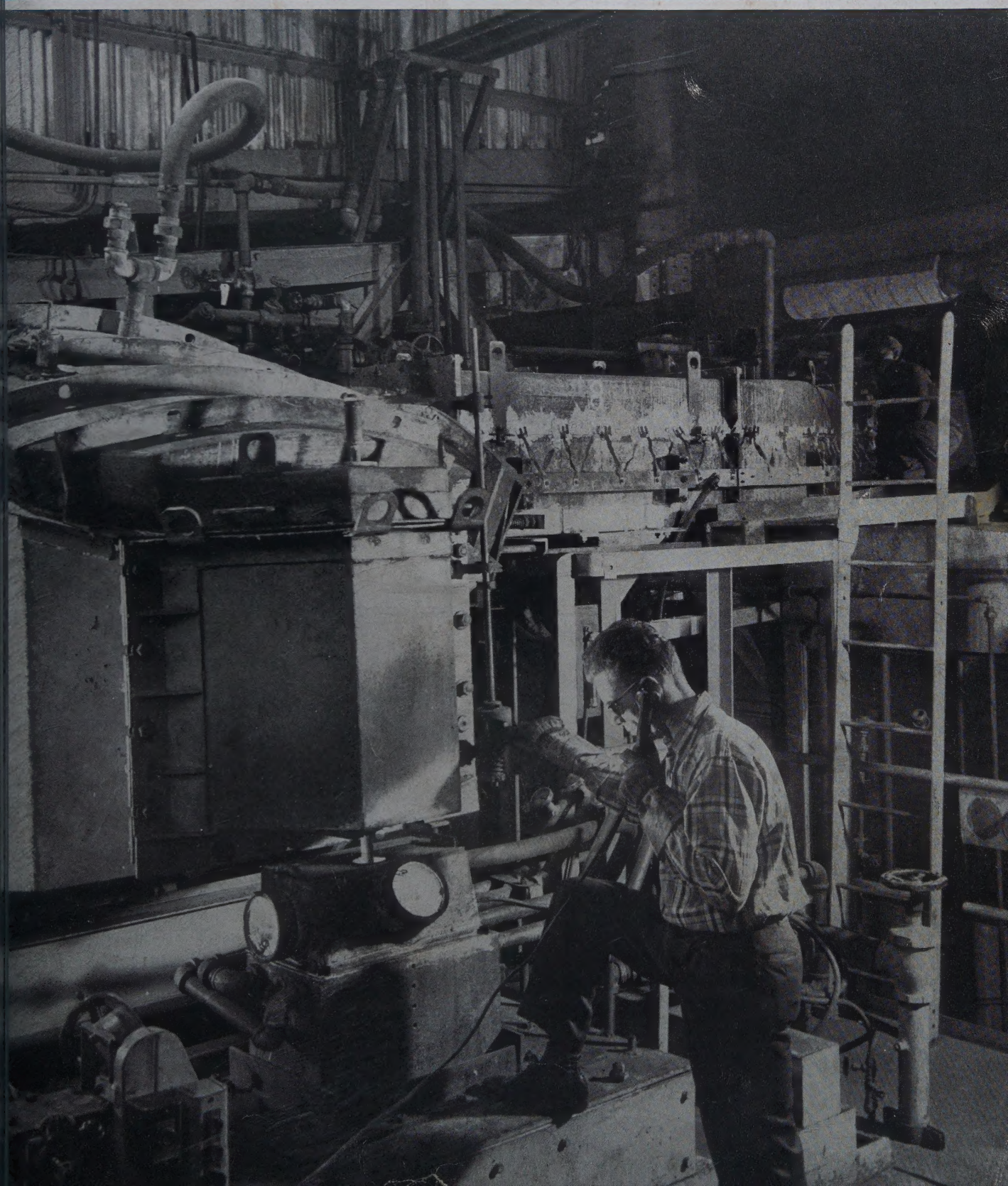


EMBER 1949

In Two Sections — Section I

*Journal of*

# METALS





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# METALS

## IN THIS ISSUE

VOL. I, NO. 12

DECEMBER 1949

### COVER

First and exclusive picture of continuous casting (Rossi type) machine at Cartaret, N. J., plant of U. S. Metals Refining Co. This is the world's only continuous casting of oxygen-free copper; some phosphorized copper has been cast and the company is experimenting with tough-pitch. Output is 100-125 tons per day in 5-8-in. rounds and 4x4-in. bars. Metal comes from a Summey rocking furnace down the Drycolene-gas filled electrically heated launders into the induction furnace over the mold chamber.

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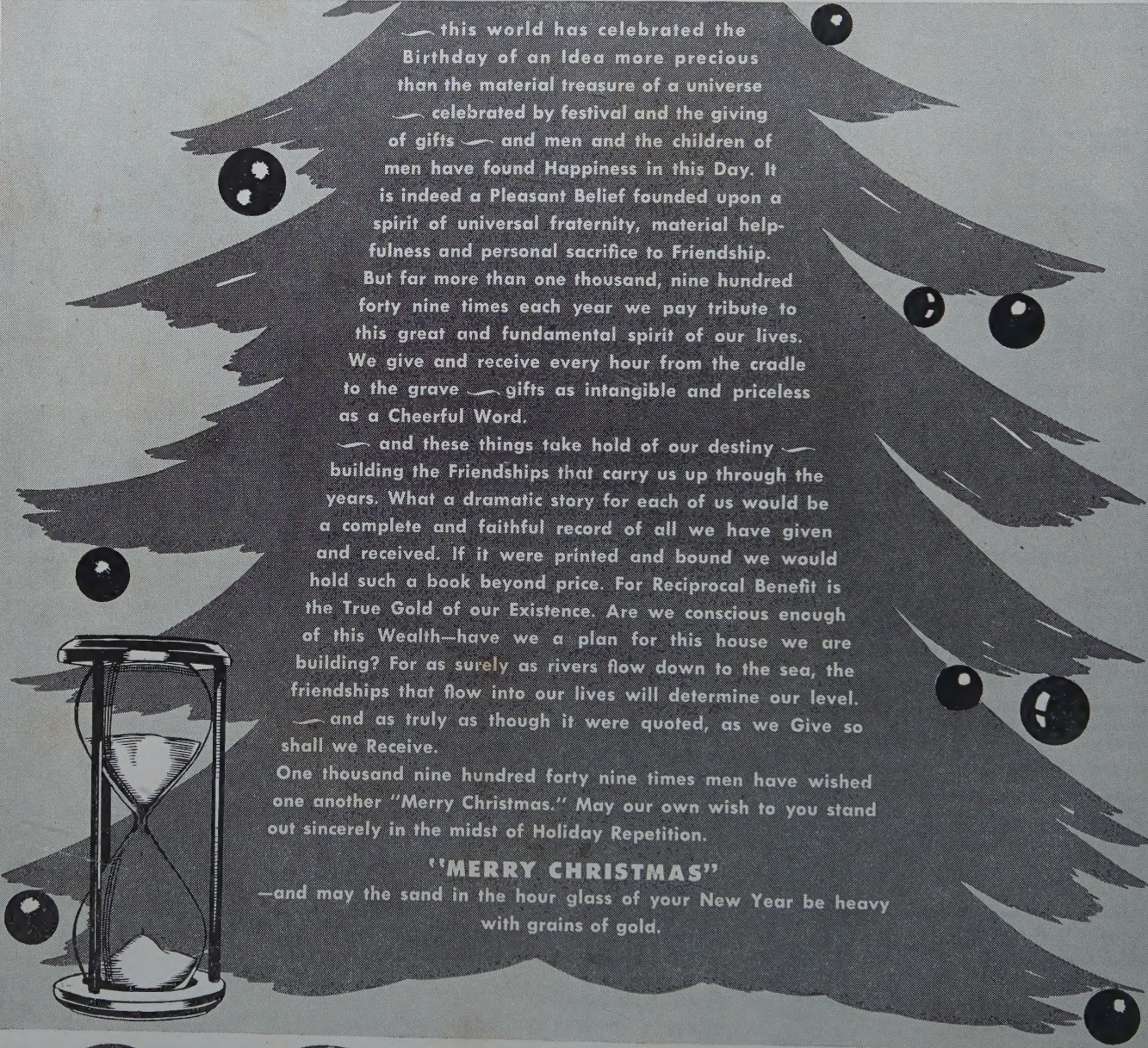
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# One Thousand, Nine Hundred Forty Nine Times



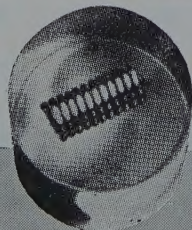
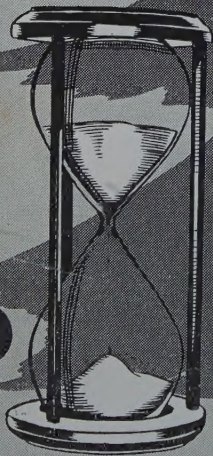
— this world has celebrated the Birthday of an Idea more precious than the material treasure of a universe — celebrated by festival and the giving of gifts — and men and the children of men have found Happiness in this Day. It is indeed a Pleasant Belief founded upon a spirit of universal fraternity, material helpfulness and personal sacrifice to Friendship. But far more than one thousand, nine hundred forty nine times each year we pay tribute to this great and fundamental spirit of our lives. We give and receive every hour from the cradle to the grave — gifts as intangible and priceless as a Cheerful Word.

— and these things take hold of our destiny — building the Friendships that carry us up through the years. What a dramatic story for each of us would be a complete and faithful record of all we have given and received. If it were printed and bound we would hold such a book beyond price. For Reciprocal Benefit is the True Gold of our Existence. Are we conscious enough of this Wealth—have we a plan for this house we are building? For as surely as rivers flow down to the sea, the friendships that flow into our lives will determine our level. — and as truly as though it were quoted, as we Give so shall we Receive.

One thousand nine hundred forty nine times men have wished one another "Merry Christmas." May our own wish to you stand out sincerely in the midst of Holiday Repetition.

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—and may the sand in the hour glass of your New Year be heavy with grains of gold.



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**R. M. Bayle** (p. 15): Born Sawyer City, Pa., attended high school in Ambridge, Pa., graduated from Ohio Northern University with degree of B.S. in E.E. His home is in Chicago. Mr. Bayle has been continuously associated with Westinghouse Electric Corp. since 1918, Electrical Test, Pittsburgh, Service Dept., General Engineering, Consulting and Application Engr. . . . **Arthur B. Wilder** (p. 20): See November issue Journal of Metals. . . . **Harmon E. Keyes** (p. 29):

Born Florence, Wis., attended Lincoln high school in Seattle, Wash. Went to M.I.T. and University of Washington, graduating with degrees of B.S., M.S.-Ch.E., Ch.E. (Prof.). Is a member of AIME and now lives in Phoenix, Ariz. He has worked for the Consolidated Mining & Smelting Co., of Canada, as Assistant Chemist; Bunker Hill & Sullivan, U. S. Bureau of Mines; Pinto Valley Co., Miami Copper Co., and General Engineering Co. Has been a Consultant for Carnegie Illinois Steel Corp. He was a Major in the Chemical Warfare Service in World War II and has a commission as Lt. Col. Chemical Corps. Besides his reserve officer activities his major recreation is fishing. . . . **S. S. Cole** (p. 905): Born in Cuba, N. Y., attended Hornell, N. Y., high school, and graduated from Alfred University, Pennsylvania State College with B.Sc., Ph.D. degrees. Is a member of AIME and lives in Metuchen, N. J. Was on the staff of Mellon Institute for some years, now Research Chemist and Asst. Manager of Research of National Lead Co., Titanium Division. Mr. Cole's chief recreations are travel and investigations of minerals and gems. . . . **H. Sigurdson** (p. 905): Born in Winnipeg, Canada, and graduated from University of Manitoba with B.Sc. degree. For some time connected with Defense Industries, Ltd., Montreal, the British-American Oil Co., Toronto, as Development Engineer during the early stages of the war and later becoming Chemist. Mr. Sigurdson lives in Metuchen, N. J., and is on the staff of National Lead Co., National Lead Co. Titanium Div., South Amboy, N. J. He enjoys outdoor activities such as tennis, skating, swimming, etc. . . . **D. L. Armant** (p. 909): Born in Binghamton, N. Y., attended high school in Johnson City, N. Y., and graduated from Alfred University with B.S.-Ceramic Eng. degree. Now lives in Fanwood, N. J. Was on the research staff of National Lead Co., Titanium Div., until he was obliged to take time out for service as 1st Lt. in the U. S. Army Air Forces in the Mediterranean Theatre, but he has again resumed his duties at the National Lead Co. Mr. Armant's interests range from photography, music and gardening to woodworking and sports. . . . **Chas. H. Moore, Jr.** (p. 914): Born in New Market, Va., attending high school there and graduated from University of Virginia with B.S.-M.S. degrees and Ph.D. from Cornell University. Is a member of AIME and his home is in Plainfield, N. J. Taught Mineralogy in Cornell, later becoming Asst. Prof. of Mineralogy in Penn State; was Petrographer, Research Lab., Carborundum Co., Dept. Supervisor, Research Lab., Titanium Div., National Lead Co.,

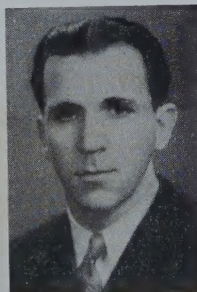


H. E. Keyes

now is Assistant to Research Director there. Mr. Moore has previously presented an AIME paper on Formation and Properties of Single Crystals of Synthetic Rutile. His chief recreations are fishing and golf. . . . **T. E. Tietz** (p. 921): Born in Pittsburgh, and attended high school in Los Angeles, graduating from University of California with degree of B.S. in Mechanical Engineering. He is a Student Associate member of AIME and lives in Berkeley, Calif. Was Design Engineer with Lane-Wells Co., Los Angeles, and currently is Research Engineer at University of Calif. His chief activities are tennis and gardening. . . . **L. J. Klingler** (p. 927): Born in Canton, Ohio, attended McKinley high school there and graduated from Case Institute of Technology with B.S. and M.S.-Phys.Met. degrees. His home is in Wickliffe, Ohio. Worked at Westinghouse Electric Corp. as Materials Engineer; served in the U. S. Navy during the war and is now on the staff of Case Institute Metals Research Lab. Mr. Klingler is interested in photography and badminton is his chief sport. . . . **A. G. Guy** (p. 933): See September issue Journal of Metals. . . . **John L. Lamont** (p. 957): Born in Malden, Mass., attended high school in Allentown, Pa., graduating from Lehigh University. Now lives in Niagara Falls, N. Y. Is Research Metallurgist at Union Carbide and Carbon Co., Niagara Falls, N. Y. Mr. Lamont has previously presented AIME papers. Mr. Lamont is a stamp collector and enjoys golf and fishing. . . . **Walter Crafts** (p. 957): Born in Oberlin, Ohio, attending high school there and graduated from Upper Canada College, Toronto, Ont., and Yale University with B.A. degree, and M.S. degree from M.I.T. Is a member of AIME and lives in Niagara Falls, N. Y. Mr. Crafts has previously presented several AIME papers. Golf and fishing are his chief recreations. . . . **Thos. E. Leontis** (p. 968): Born in Plainfield, N. J. Attended Plainfield high school, graduating from Stevens Inst. of Technology and Carnegie Tech with M.E., M.S. and D.Sc. degrees. Is a Junior Member AIME and lives in Midland, Mich. He was Instructor at Stephens Inst. for a time, becoming Metallurgist at Vanadium Corp. of America; was Dow Chem. Co. Graduate Fellow at Carnegie Inst. of Tech., and is currently Metallurgical Engineer with Dow Chemical Co. Magnesium Lab. Mr. Leontis has previously presented papers on Rates of High Temp. Oxidation of Mg and Mg Alloys; Prop. of Ce—Containing Mg Alloys at Room and Elev. Temp.; Room and Elev. Temp. Prop. of Some Sand Cast Mg Alloys Containing Zn. . . . **Paul Pietrokowsky** (p. 948): Born in Los Angeles, attended high school there, went to U. C. L. A. and received degrees of B.S.-Mech. Engr. and M.S.-Phys. Metall. from University of California. Is a Junior



L. J. Klingler



T. E. Leontis

Member AIME and lives in Midland, Mich. He was Instructor at Stephens Inst. for a time, becoming Metallurgist at Vanadium Corp. of America; was Dow Chem. Co. Graduate Fellow at Carnegie Inst. of Tech., and is currently Metallurgical Engineer with Dow Chemical Co. Magnesium Lab. Mr. Leontis has previously presented papers on Rates of High Temp. Oxidation of Mg and Mg Alloys; Prop. of Ce—Containing Mg Alloys at Room and Elev. Temp.; Room and Elev. Temp. Prop. of Some Sand Cast Mg Alloys Containing Zn. . . . **Paul Pietrokowsky** (p. 948): Born in Los Angeles, attended high school there, went to U. C. L. A. and received degrees of B.S.-Mech. Engr. and M.S.-Phys. Metall. from University of California. Is a Junior



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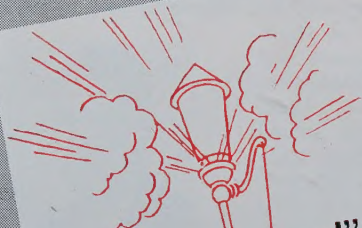
P. Pietrokowsky

(Continued on Page 7)



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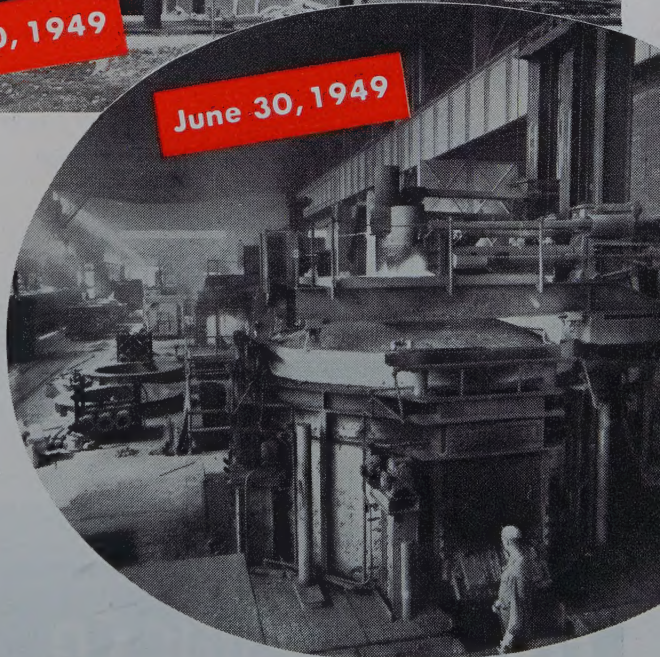


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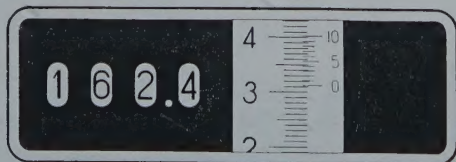
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A. K. Schellinger

tended Maine Jr. College, Park Ridge, Ill., and received B.A. and M.S.-Met. Eng. degrees from Stanford University, Calif. Is a Student Associate member of AIME and lives in Vallejo, Calif. Served as Lt. (jg) U. S. Navy and is now Metallurgist at the Selby Plant, American Smelting & Refining Co. Lt. Olsen's chief activity is in connection with Organized Naval Reserve. . . .

**A. Kenneth Schellinger** (p. 984): Born in Dawson Y. T., Canada, attending high school in Palo Alto and graduating from Stanford University with A.B., M.S., degrees. Is a member of AIME and his home is in Palo Alto, Calif. Was associated with Selby Plant, A. S. & R. Co. as Junior Metallurgist; subsequently Metallurgist and now Chief Chemist of Andes Copper Mining Co., Potrerillos, Chile S.A. Mr. Schellinger has previously presented an AIME paper on Simultaneous Grinding and Flotation. . . .

**John C. Redmond** (p. 987): Born in Deerfield (Utica), N. Y., and attended Townsend Harris high school in New York City, graduating from Hamilton College, Clinton, N. Y., and George Washington University, Washington, D. C., with B.S. and A.M. degrees. Is a member of AIME and he lives in Greensboro, Pa. Mr. Redmond was associated with National Bureau of Standards as Junior Chemist, becoming Chemist at Battelle Memorial Institute; Research Chemist and Supervisor of Chemical Processes at Youngstown Sheet & Tube, and is now Research Engineer and Chief Chemist and Research Director of Kennametal, Inc., Greensboro, Pa. His hobby is model railroading. . . .

**E. N. Smith** (p. 987): Born in Galeton, Pa., and attended high school in Punxsutawney, Pa., graduating from Pennsylvania State College with degree of B.S.-Chem. Eng. His home is in Ligonier, Pa. He was on the staff of Republic Steel Corp., Youngstown, as Chemist; later Process Chemist of Youngstown Sheet & Tube, and subsequently Chemical Engineer in the Office of Chief of Ordnance, Washington, D. C. Mr. Smith's chief recreation is music and golf. . . .

**E. W. Palmer** (p. 995): Born in Johnson City, N. Y. and attended Unadilla, N. Y., high school, graduating from Rensselaer Polytechnic Institute with Ch.E. degree. Is a member of AIME and lives in Watertown, Conn. Has been on the staff of American Brass Co. since graduation, and for the past five years Research Metallurgist there, Photography is his hobby. . . .

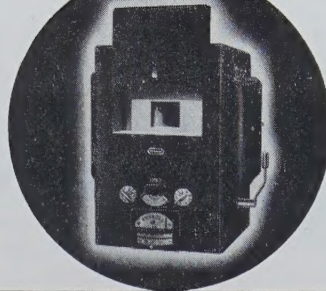
**F. H. Wilson** (p. 995): Born in Kuling, Kiangsi, China, and attended high school in Princeton, N. J. Has degrees of B.S., M.A., M.Eng., D.Eng., from Princeton, Wesleyan and Yale Universities. Is now connected with the Technical Dept., American Brass Co.

Member of AIME and lives in Berkeley, Calif. Served in U. S.-A.A.F. as Mechanic and Engineering Officer, was Design Engineer with Futurecraft Corp., Montrose, Calif., and currently is Research Metallurgist and Engineer for ONR research project, University of Calif. Mr. Pietrowsky's chief recreation is watercolor painting and mountaineering. . . .

**Robert P. Olsen** (p. 984): Born in Chicago and went to Lane Technical high school there. Attended



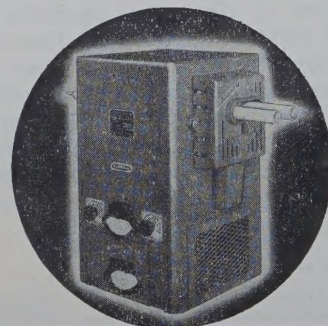
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## SECTION ACTIVITIES

### Rio de Janeiro Section

According to Emerson I. Brown, the local section AIME has recently held two meetings, one on Sept. 22 and the second on Oct. 13. At the Sept. 22 meeting, Dr. Luciano Jacques de Moraes, director of Companhia Aços Especiais de Itabira gave a resumé of the activities and ultimate objectives of this company which was formed in October, 1944. This will be an integrated plant for the production of 53,000 tons per year of alloy steels and alloy steel products, beginning with mining the iron and manganese ore and producing charcoal from the company's property. Not only the reduction and steel-making facilities, but also the casting, forging and machining operations can be carried out at this plant which is not yet complete at Coronel Fabriciano, Minas Gerais.

The blast furnace is in operation; it has a designed capacity of 200 tons of pig iron per day with charcoal fuel and 350 tons if coke is used. Much of the steel-making and other equipment for completion of the plant is on hand. The consulting engineers, H. A. Brassert & Co. have provided for considerable flexibility in the steel-making process and for a wide range in types of alloy steel products to be made. The mineral rights controlled by this company formerly belonged to the Brazilian Iron & Steel Co., and have been examined by the well-known American geologists, C. K. Leith and E. C. Harder.

A hydro-electric plant 28 km from the steel plant is under construction; in its initial phase, it will produce 14,000 kw from a diversion of the Piracicaba

River through a head of 114 m. The volume of water is said to be ample for the eventual addition of two more units.

At the subsequent meeting of the Rio local section on Oct. 13, Dr. Sylvio Frôes Abreu, who had just returned from the U. S. as official Brazilian delegate to the UNSCCUR at Lake Success, N. Y., and who also visited the U. S. Bureau of Mines' oil-shale demonstration mine and plant at Rifle, Col., gave the local section an interesting account of those events. A personal report by a competent observer such as Dr. Frôes Abreu was of great interest and value to the Rio local section. A summary of Dr. Frôes Abreu's remarks has been distributed to the members of this local section, but there is no point in relating them here.

### New York Section

The installation of a ladies' night proved so successful last year that the Nov. 10 meeting this year was scheduled as ladies' night, according to T. B. Counselman. The decision was an excellent one, in that this year's meeting was even more successful than last. Over 100 were served at the dinner, of which a large proportion were ladies.

Although the meeting was an off-the-record meeting, no secrets were divulged, but the subject "Nuclear Energy—Present and Future," was certainly explained in a different way. Dr. Clark Goodman, Associate Professor of Physics of the Massachusetts Institute of Technology, was the speaker. Referring to the Smythe report and other declassified information he used analogies and everyday language to explain a subject that is difficult to understand even by those working in related fields, let alone those whose only knowledge of the reactions in the atomic bomb has been gathered through the daily press. Suffice it to say that Dr. Goodman brought a subject which was extremely timely, due to the recent announcement that the Russians have the

C. D. King, Chairman (center), Iron and Steel Division, AIME, George Enzian (right), Secretary of the Pittsburgh Section of AIME, and George Donaldson, Vice-Chairman of the Pittsburgh Section of the AIME, congratulate Henry Warren, Jr. (left), on his election to the Chairmanship of the Pittsburgh Section of the National Open Hearth Committee of AIME following the all day meeting of the Pittsburgh Section, AIME, at which a record attendance of 794 was set on Oct. 28.





atomic bomb, down to the level where it could be understood and appreciated by those in attendance.

### Southwestern Section

Chairman A. H. Sommer reports that one of the most successful meetings of the Section National Open Hearth Committee was held at the President Hotel in Kansas City, Oct. 14, with 102 persons attending. The Sheffield Steel Co. very capably

(Continued on p. 32)

## Engineering Societies Service

THE following employment items are made available to AIME members on a nonprofit basis by the Engineering Societies Personnel Service, Inc., operating in cooperation with the Four Founder Societies. Local offices of the Personnel Service are at 8 W. 40th St., New York 18; 100 Farnsworth Ave., Detroit; 57 Post St., San Francisco, 84 E. Randolph St., Chicago 1. Applicants should address all mail to the proper key numbers in care of the New York office, and include 6¢ in stamps for forwarding and returning application. The applicant agrees, if placed in a position by means of the Service to pay the placement fee listed by the Service. AIME members may secure a weekly bulletin of positions available for \$3.50 a quarter or \$12 a year.

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- \* Blast furnace slag, reduced to 12-16 mesh, is being used to replace silica sand for annealing-oven seals at the Armco plants. Armco is also using the granulated slag to replace silica sand for sanding track in its Mont coal mines and is experimenting with the slag for rock dusting. In both instances the slag is fully as good as the sand--far cheaper--and, of course, avoids the silicosis hazard.
- \* Some electric furnace operators may be able to adjust arc current for optimum results, but automatic equipment is far better. R. M. Bayle (p. 14) describes Optimelt automatic recalibrating control which results in improved refractory life, lower electrode consumption and duplication of operation from heat to heat.
- \* Oxygen-enriched blast, end-point evaluation and temperature control are included in A. B. Wilder's (p. 20) appraisal of the newest developments in bessemer converter operation.
- \* The French are fearful of German steel competition, but recent meetings in Paris between the heads of French Association for Economic Relations with Germany (AFREA) developed some discussion of a plan that might satisfy both countries.
- \* Vereinigte Stahlwerke, the Ruhr steel trust, could increase its share capital, selling three-quarters of the new capital to France (which would invest American money indirectly), the United States and Britain. This would, supposedly, guarantee security against rearmament and, incidentally, against excessive competition once the protection of quotas is gone.
- \* Fifteen plants comprising the Acid Openhearth Association have granted \$20,000 to the University of Pittsburgh to renew a research program on refining problems. Officers of the Association are C. N. Arnold, president of Blaw Knox; H. F. Dowie, vice-president, Mesta; F. C. T. Daniels, secretary, Mackintosh-Hemphill; and A. R. Altman, treasurer, Heppenstall Co.
- \* Cemented titanium-carbide compositions prepared by powder metallurgy methods show transverse rupture strengths up to 175,000 psi, or 75 pct greater than previously reported (p. 987). Cemented titanium-carbide compositions offer excellent possibilities as high-temperature materials.
- \* Ilmenites having 38-44 pct  $TiO_2$  have been smelted in the laboratory to produce fluid slags with 65-69 pct  $TiO_2$ ; the final slag requires 5-11 pct  $MgO$  and 9-13 pct  $CaO$  in order to have desired fluidity (p. 909).
- \* Various rare-earth metals enhance the hardness, strength and creep resistance of magnesium at room and elevated temperatures (p. 968).
- \* Long before uranium deposits became a state secret, the Great Soviet Encyclopedia (1936) Vol. 56, pp. 245-47 described in detail Russia's deposits which spread from Samarkand eastward some 300 miles to Tuya Muyan, with a number of ores up to 80 pct uranium content. Atomgrad is probably in this area of the old Tamerlane empire, protected by the glaciers and peaks of the Tiau-Shan and Pamir-Alai mountains, rather than where most newspapers prefer to locate it near the Turkish border in Armenia.

In 1936 only 35 pct of the Soviet Union had been explored by geologists whereas now almost 78 pct of the country has been surveyed, so that there is great likelihood that many new deposits have been uncovered.



# It's Everyone's Business

NOV. 30—Amid general public apathy, the steel industry early in the month struck its colors and met Mr. Murray on his own terms with as good grace as the trying situation permitted. Now that the steel worker had his security packet there soon were little straws in the wind that the steel consumer wouldn't for long avoid paying for the packet. Mr. Lewis, on the other hand, failed in his attempts to divide the coal owners and thus conquer, but hastily got one foot temporarily back on the ground before that man in Blair House took another swing at him. With both the steel and coal workers back on the job, blood again circulated freely through the cramped economy although it was apparent that the business structure had suffered little more than a temporary setback. The Secretary of Commerce's prediction of 2,000,000 unemployed by Nov. 1 never materialized and most pundits switched midstream from predictions of disaster to heady promises of new records of achievement.

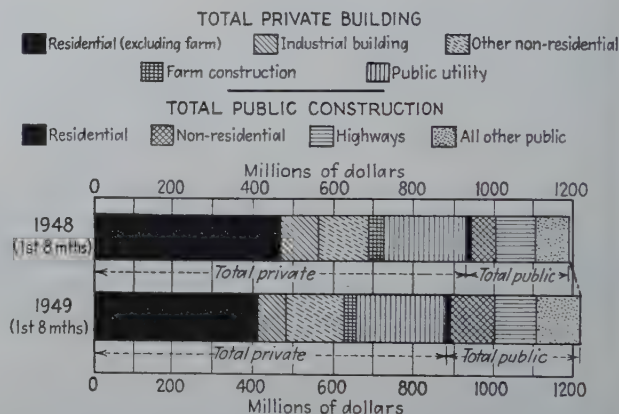
Mr. Murray walked out of the arena with a whole fistful of new laurels and with the principle of non-contributory pensions firmly implanted in the nation's economy and thinking. For the first time in many a year, Mr. Murray looked Mr. Lewis straight in the eye—and, in fact, the look tended at times to be one of benign condescension—for the old master himself was backed off into a weaker bargaining position than has been his lot since away back in the grim organizational days.

Of course, Mr. Murray had a little tidying up to do to recapture some 1,000,000 left-wing CIO workers to add to his loyal 6,000,000, but his triumphs were such that the job appeared far easier than would have been guessed a year ago. The 600,000 United Mine Workers already had their hands well into the pension grab bag, and it would seem only a matter of time until the other 9.15 million organized workers (7.2 million AFL, 1.5 million independent unions and 450,000 Railway Brotherhoods) would be fumbling around in the same bag. But for a while the 47 million unorganized workers could look forward to little more than the relatively meager crumbs from Federal Social Security. Agitation on their part to increase these payments is bound to take on a little more bite, and businessmen may also soon be tempted to lessen their opposition to increases in Social Security, as it could be a means to transfer part of industry's load onto Federal pensions.

The Federal Reserve Board's index of industrial activity fell in October to the lowest point since 1946 but did a rapid about-face in mid-November. Whether the index will continue climbing is a matter of debate. Buyers are not showing their usual Fall enthusiasm to clean off store shelves and if they continue to drag their feet the soaring index line will flatten soon. In January, ex-servicemen, who carried war insurance, will be the recipients of some \$3 billions which should offset the post-Christmas buying blues. The newest Federal Reserve Board study indicates that the automobile industry should in 1950 equal 1949 records and Detroit is already licking its chops, and understand-

ably so. For in the third quarter of 1949 the automakers stocked up profits 60 pct higher than for the same period in 1948, whereas other leading corporations suffered an 18 pct decline. General Motors distributed \$190 million, the largest lump of cash ever paid out at one time in cash dividends by an American company.

Construction promises in 1949 to break the 1925 record, with some 950,000 houses started in the year. Early last Spring it looked as if the three-year boom was beginning to stagger, but Congress kept open the golden stream of Federal credit in the form of insurance of private mortgages, and has, in fact, extended the arrangement until March 1950. Builders have been paying more attention to the almost-forgotten middle-class market, prices have dropped slightly and the consumer is quite convinced that further significant drops are unlikely. The accompanying graph indicates that industrial building has eased off slightly but this gap was more than offset by a surge of long-deferred public construction.



While the average citizen is quite willing to be dazzled in 1950 by an ever-mounting gush of consumer goods, there is still a residue of caution among those who remember the 1929 optimism and what followed. But prophets persist in their glowing estimates of the times to come. Mr. Moulton, head of Brookings Institute, has just announced his estimate of a living standard eight times higher within the next 100 years. Emil Schram, president of the N. Y. Stock Exchange, promises a tomorrow far more fruitful than the past or present. Sumner Slichter, Harvard's renowned economist "suggests" a doubled standard of living by 1980, and Donald Woodward of Mutual Life Insurance Co. says that today's \$5800 family will have \$7000 to spend in 1975. For those who won't live so long *Life Magazine* and the Twentieth Century Fund glowingly announce a golden decade ahead. With all these promises the average citizen no longer needs to trudge the streets with a frown on his face and uncertainty in his heart, and can look forward to two television sets where only one sprouted before.

Base metal prices in November were subjected to a variety of conflicting factors, and as yet long-term



demand, with the exception of copper, is a matter of day-to-day conjecture. Tied as it is to the steel economy, zinc has shown a modest recovery. The month opened with a  $\frac{1}{4}\phi$  rise to 9.75 $\phi$  a lb and by Nov. 9 some smelters were quoting 10 $\phi$  but by Nov. 14 the metal had settled back to 9.75 $\phi$ . Copper, which remained firm all during the strike period at 17.62 $\phi$  early in the month moved progressively up to 18.50 $\phi$  a lb. On the other hand, lead picked up no strength from the steel settlement but weakened. Opening the month at 13 $\phi$  a lb it fell off on Nov. 10, 16 and 21 to a current level of 12 $\phi$ .

In tin, however, it was a rat race with the market going through a series of dramatic convulsions and prices all on the downward side. The biggest holder of tin is the British Ministry of Supply and as the London Market opened Nov. 15 for the first time since 1941, the previous fixed price of £750 progressively dropped to £715 a ton. In New York the RFC, which markets the output of the Texas Smelter, persistently held to 95 $\phi$  a lb, but as British shipments approached New York the price dropped first to 85 $\phi$  then to 83 $\phi$  for spot metal. However, future metal has been selling as low as 78½ $\phi$  and heavy buying has been taking place at that figure. The market yet does not know whether the RFC will stockpile its metal or whether prices will be dropped to keep the British from capturing the market. In either case lower prices were in prospect and tinplate makers could look forward to better profits—welcome indeed to offset Mr. Murray's pension bite.

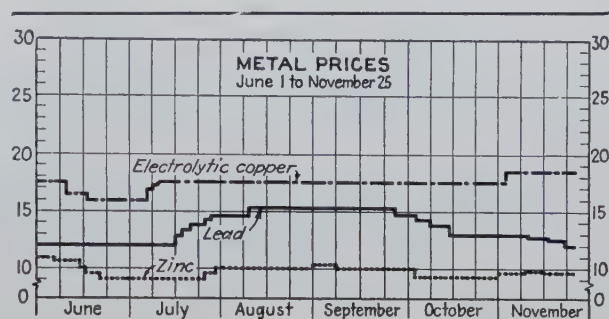
If the enamored consumer were to lift his eyes above this onrushing flood of the better life to peer across the oceans he would see England locked in preelection histrionics. Labor and Tory viewpoints on the nationalization of steel, for instance, will be carried to the hustings for the electorate to decide. It is still by no means certain that the election will be in February, and, if not—June seems the likely month. Tory policy seems to be drifting to the left but not with the conviction necessary to satisfy many British voters. Labor, on the other hand, is tired and no longer reflects any real conviction on how to solve either Britain's problems or build a better world. What the voter seems to want is a middle-of-the-road course intermixed with some real progressive and crusading idealism, but so far no offering quite fits the bill.

On the Continent it is now apparent that the Organization for European Cooperation (OEEC) can do little to curb investment in the European steel industry which may result in excess capacity by 1952-53. The OEEC has done its best but it is difficult indeed for an international body such as this to restrain the development plans of individual nations.

European steel capacity in 1950-53 is expected to reach 57.5 million tons—on the questionable assumption that German output will not exceed the current limit of 11.1 million tons. This compares with 53 million tons (including German production of 20 million tons) for prewar Europe. While the 4 million ton excess is considered by many to be of no great consequence, the "Rollman" report, yet unpublished, takes the opposite view. Mr. Rollman, head of the steel committee of ECE at Geneva, con-

cludes that 4 yr from now Europe will be producing 8 million more tons than can be sold either at home or abroad. Even this conclusion appears unrealistic as it also assumes German production of 11.1 million tons. The French are expecting an all-out German assault on this limitation soon, and it is questionable as to whether the limitation can be maintained. All the factors point toward fertile ground for the usual cartel arrangement to sprout and flourish.

Inflation seems to be on the move again in France under the impetus of a severe drought, a budget



which has again become unbalanced, and an incipient but rising discontent among workers because only a comparative few are gathering in the fruits of the better life.

Halfway around the world again, in Tokyo, there is a growing opinion among the conquerors that a peace treaty should end the state of war. The victories of Communist arms on the mainland make it desirable to give the Japanese more and more discretion in the handling of their own affairs. But to draft a peace treaty requires participation of Russia and that prospect is unnerving to most of the diplomats. It's going to take quite a bit of doing to figure out a satisfactory angle shot to satisfy the Japanese while at the same time sidestep Russian thin-edge-of-the-wedge tactics.

Southwest of Japan, the prospect of American economic aid to countries of South East Asia is beginning to appear more likely. The International Bank may soon extend a helping hand to India, the Export-Import Bank is looking with favor on the new Indonesia and sizable funds may be appropriated to support anti-Communist work "in the vicinity of China." India has the highest priority and Pandit Nehru talked tentatively about financing during his recent visit. Mr. Nehru also talked specifically about "peace," but no one was bold enough to suggest that he practice in Asia what he preached in the U. S.; for India solved the Hyderabad dispute with Pakistan by force of arms and seems equally determined to resolve the Kashmir dispute in the same manner. Meanwhile a U. S. Steel Corp. team is in Pakistan studying manufacturing and marketing problems. Pakistan's Prime Minister, Liaquat Ali Khan, however, has been affronted by Washington's failure to invite him for a visit, and has set up a date to go and talk to Stalin.

With all this, the past isolation of the United States is something difficult to recall. It seems hardly possible that less than 20 yr ago many a politician rode into office merely on the vacuous promise to make Britain pay off old war debts.



# Optimelt Automatic Recalibrating Control

for arc-furnace regulators

**F**IG 1 illustrates a typical form of the characteristic power input curves of the direct-arc furnace used in the melting and refining of steel. These characteristics have been published and analyzed in many previous papers and their general forms are familiar to most electrical engineers handling arc-furnace applications. The various designs of electric-arc furnaces of American manufacture present many variations of circuit geometry, electrical ratings and tap voltages and the actual numerical relations existing between arc current, voltages, kilovolt amperes, kilowatts and power factor may vary somewhat, yet the general shape of these curves is maintained in all cases.

It will be noted that, for any given tap voltage, the power input to the circuit increases as the electrodes are lowered to increase current, levels off and finally decreases as the arc current is further increased. Using the curves for tap No. 6, for example, the maximum circuit KW occurs at arc current (A). Following up the ordinate for arc current (A) to the power factor curve it is found that this maximum circuit KW occurs at a power factor of 70.7 pct.

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This paper is to be presented at the Seventh Annual Electric Furnace Steel Conference, Iron and Steel Div., A.I.M.E., Pittsburgh, Dec. 8-10.

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Now if the arc current is similarly plotted for maximum arc KW it will fall at current (B). Due

to the  $I^2R$  losses in the transformer, secondary bus, cables, mast arms, electrodes, etc., this will always be somewhat less than the maximum circuit KW arc current (A) and at a power factor better than 70.7 pct. (i.e., nearer unity).

This same fundamental fact can be demonstrated for the other voltage taps. In every case there is an optimum value of arc current that produces maximum arc KW for that particular secondary voltage. This value of arc current is always less and the power factor is always greater than a similar arc current and power factor for the point of maximum circuit KW for that particular voltage.

The lesson the electrical engineer draws from these fundamentals and passes on to the operating man is that there can be no sound reason for operating at an arc current beyond that which results in maximum arc KW. He merely pours additional and expensive kilowatt hours into circuit losses which do not melt steel.

He can also demonstrate that there is reduced efficiency in operating at a point much below this optimum arc-current value. Even though the power factor is higher, the actual reduction in melting heat desired can be obtained much more efficiently by going to a lower voltage tap.

It will follow that any method of capitalizing on the efficiency inherent in such ideal circuit conditions will yield the greatest savings during the period of greatest power input. This unfortunately is that selfsame period of violent regulating duty during the meltdown period. Approaching the problem in this manner, it is easily seen how utterly impossible it is



by R. M. Bayle

**O**PERATION at the optimum arc-current value might be within the range of ability of some furnace operators, but for most the resort to supplementary regulator supervisory equipment, such as the Optimelt control, will assure a whole host of advantages, such as less KW-hours per ton, less time per heat, improvement in refractory life, reduced consumption of electrodes and consistent duplication of operation from heat to heat.

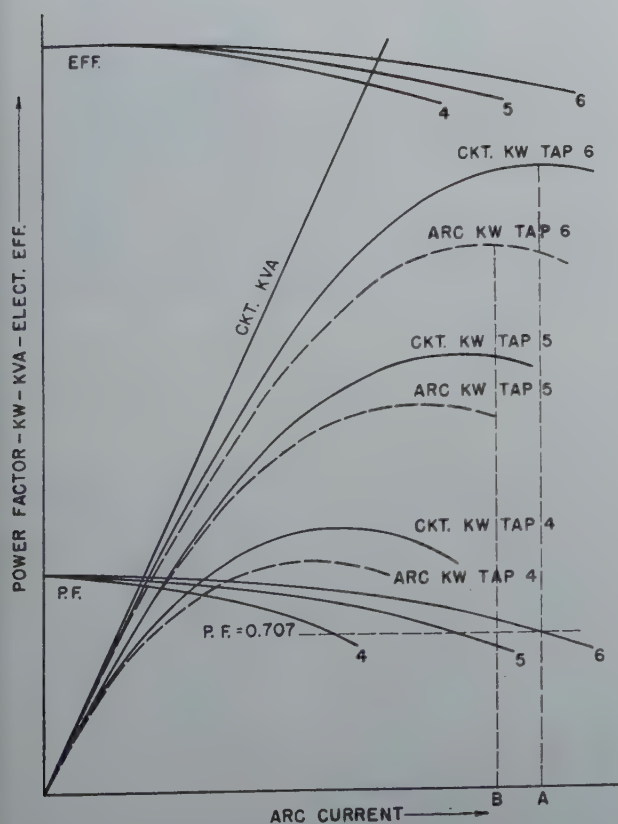


Fig. 1—Typical characteristics of an arc-furnace circuit, after E. W. Browning.

for any operator to observe his dancing instruments, and manually set the current adjusting rheostats in each of the three phases of an arc furnace regulator to secure anything approaching a uniformly efficient condition. Granting that some few operators did get "the feel" of it there is never any assurance that every operator on every turn, day in and day out would maintain these optimum conditions.

The Optimelt control built by the Westinghouse Electric Corp. is a supplementary regulator supervisory equipment which will function automatically and definitely assure the optimum operating conditions described above. This is the control equipment applied to the arc-furnace regulators of the South Chicago plant of the Republic Steel Corp. and which produced the remarkable record of improved operating efficiency described in the companion paper by Mr. Wynd which is to be presented to the Conference. (See also *Journal of Metals*, January 1950.)

The electrical equipment supplied for an Optimelt control consists of two basic forms, depending on whether the application is to be made to existing equipment or to a completely new arc-furnace installation.

**Optimelt Control for Existing Installations:** Fig 2 illustrates a standard auxiliary panel for existing installations, which contains all the necessary equipment for controlling optimum conditions on five voltage taps. This panel is usually mounted on the rear of the operator's panel, as all the circuit wiring, which will be changed, terminates at this location. However, the panel can be mounted elsewhere if a



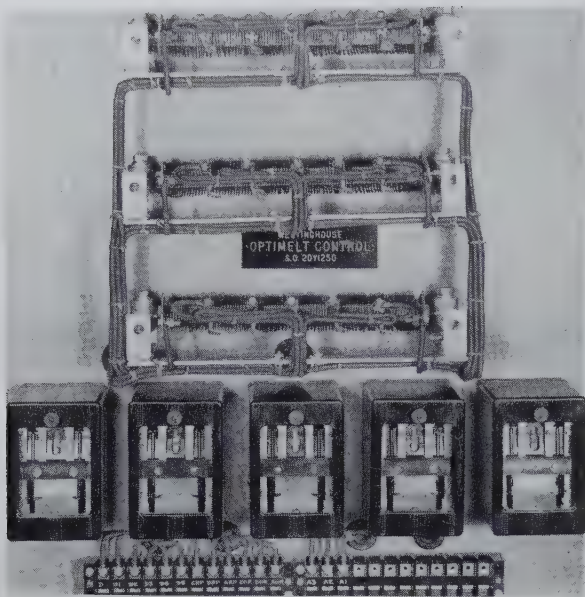


Fig. 2—Front view of Optimelt control panel.

remote location is preferred and such advantages outweigh the extra wiring runs which will be required.

The five relays located across the bottom of the panel are standard six pole switchboard relays. The coil of each relay is wired in parallel with the corresponding arc-furnace regulator voltage relay for each tap. Consequently, two relays (one new Optimelt control relay and one existing arc-furnace regulator relay) close on each tap position and one set of existing furnace tap-changer auxiliary contacts can therefore serve both functions. Each relay has a set of silver contacts for the functions of each of the three phases of the regulator, and hence the tap selection and subsequent relay operation will simultaneously present the optimum current settings for all three phases of the regulator.

In the upper-middle section of the panel are mounted three tapped edgewound resistor tubes. One resistor is provided for each phase of the regulator and the optimum current selection for each of the controlled voltage taps on each of the three phases can be made on these tubes.

Fig. 3 shows an actual installation view of the Republic Steel Corp., South Chicago Plant units. The left-hand view shows the Optimelt panel swung out to permit access to the wiring. This illustrates the details of the wiring and shows the fixed shunting resistors. The right-hand view shows the Optimelt control panel in the closed and normal position. This illustrates the adjustable shunting resistors for each phase and each tap and also the auxiliary relays for each tap. Eight panels of this type were manufactured for this plant, although only five operated on Optimelt control during the test period covered by Mr. Wynd's paper. These panels are hinged to the rear framework of the operator's panel. The relays are mounted on the front of the panel and the adjusting resistors are mounted on the rear of this same panel. Only three melting taps are controlled in this application and hence only three relays are needed. The resistor assembly, in this case, was

made with vitreous enameled slider tubes. The adjustable taps were put on separate tubes to facilitate the ease and precision of the adjustments.

**Optimelt Control for New Installations:** For applications involving a completely new furnace the Optimelt control can be further simplified by the use of a separate auxiliary switching unit built into and operated by the furnace transformer tap-changer mechanism. Such a unit is shown on fig 4. These switches perform the same functions as the relays previously described for the application of Optimelt control to existing installations.

The resistors for adjusting the optimum current conditions on each voltage tap are then made a part of the main regulator panel. Fig 5 shows a front

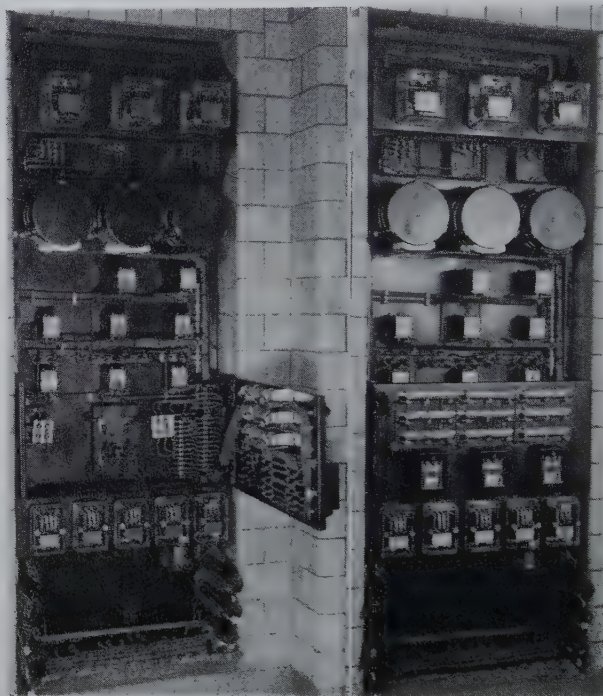


Fig. 3—Optimelt control panel installed at Republic's South Chicago plant. At left the panel is swung out to permit access to wiring; at right the panel is closed and in normal position.

view of a standard Rototrol arc-furnace regulator panel. The three phases are arranged in duplicate horizontal sections. The tapped edgewound resistor in the upper right-hand corner of each section is the Optimelt control resistor.

**Electrical Circuits:** A simplified schematic diagram of a representative Optimelt control circuit is shown in fig 6. This arrangement is drawn to show the control of three melting and three refining voltage taps, and is perhaps the maximum control any operator would find practical to use. Only one phase of the current regulating portion of the arc-furnace regulator is shown. While all arc-furnace regulators have voltage elements, in this particular case the concern is with current adjustments only.

First ignore the adjusting network between points CD and G. If this were absent, the full output of the current transformer of this phase would flow through the ammeter and through wire CD to the current element of the regulator and thence to the ground and return wire G. In all arc-furnace regu-



lating systems the regulator requires a fixed current to supply the ampere turns which balance the voltage element and satisfies the regulator. With the above-assumed circuit the electrode current needed to supply the required balancing current would be a minimum, as all of the current from the electrode current transformer secondary goes directly into the current element of the regulator.

In the conventional hand-adjusted regulator system an adjusting rheostat is added with a fixed section (CO-CF) and a variable section (CF-G) connected in parallel with the current element at points CD and G. The fixed section sets the maximum amount of current that can be shunted away from the current element when the variable element of the rheostat is in the "all out" or minimum resistance position. This is the maximum electrode current setting, as the main electrode current must build up to a value which will supply both the fixed current required to satisfy the regulator and also the maximum current passing through the shunting portion

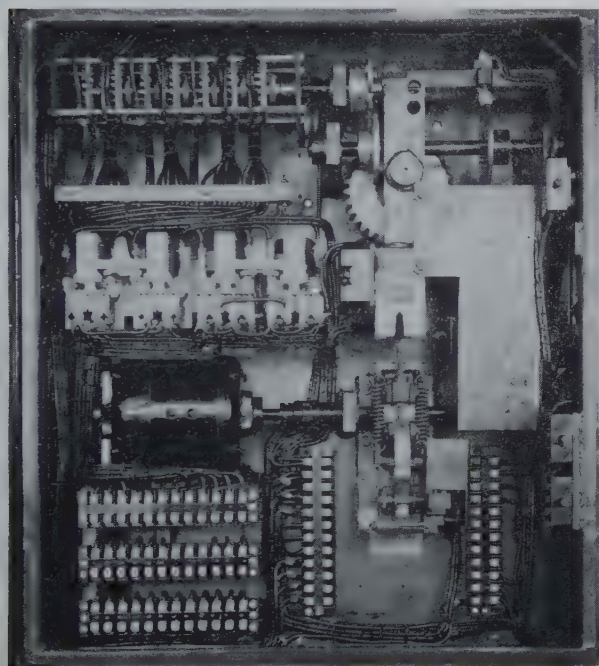


Fig. 4—Bank of auxiliary switches which can be attached to and operated by a furnace transformer tap-changer mechanism.

of the circuit. Then as the operator moves the variable portion of the rheostat towards the "all-in" or maximum resistance point, the shunted current will decrease and the electrode current needed to balance the regulator will also decrease. And so the adjustable range of the arc-furnace regulator depends on the resistance shunted across the current element. Maximum resistance means minimum regulated current and conversely minimum resistance means maximum regulated current.

When Optimelt control is added, an additional tapped and adjustable resistor (CD-CO) is added as shown in fig. 6. Assuming a desire to operate on melting tap No. 5, two contacts will be closed by the action of the tap-changer mechanism, either

by direct switching or by a relay operation. The first contact (5) shorts the variable portion of the rheostat and the other contact connected to wire C5 shorts a portion of the Optimelt resistor. Therefore, there is a fixed resistance (CO CF) and preset fixed resistance (C5-CD) in the shunting circuit. Resistor tap C5 is set by test to give the maximum arc KW on this particular voltage tap. As the adjusting rheostat is shorted, the operator has no control on this tap and maximum melting KW is automatically set up every time the tap changer is moved to this point. Similar optimum settings are made for the other melting taps Nos. 4 and 6.

When the refining taps Nos. 1, 2 or 3 are used, a slightly different circuit is set up. Here flexibility of adjustment is necessary and the variable portion of the current adjusting rheostat is left in the circuit. The adjustments made on resistor taps C1, C2 and C3 will be to set each resistor tap with the rheostat in the maximum arc-current position so that the regulated current can never reach a high enough value to impair the charge analysis by carbon pickup. The operator should always keep in mind that Optimelt control on the refining taps is primarily concerned with limiting of the current to a metallurgically safe value and not with power savings.

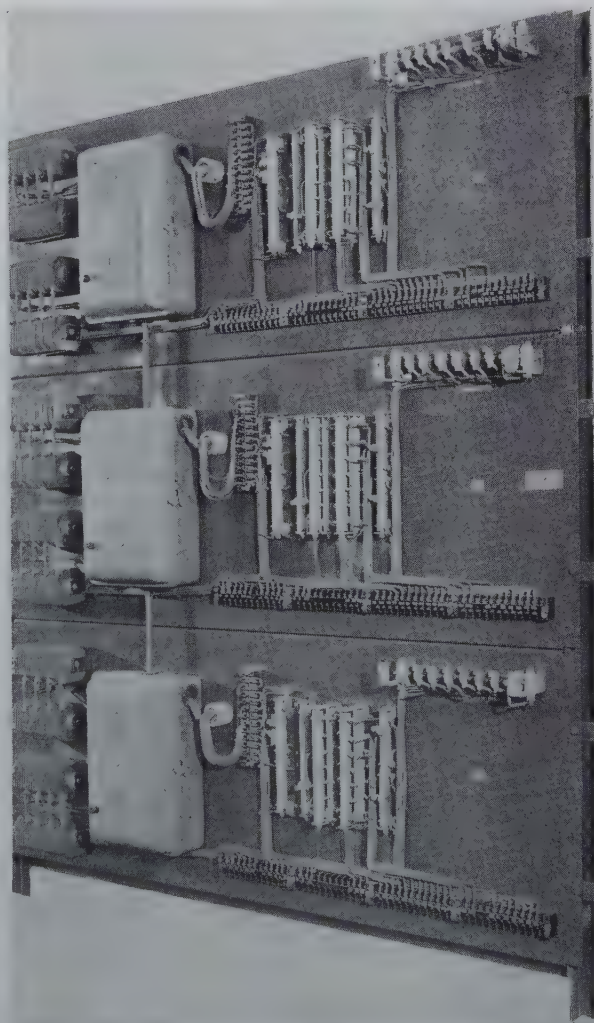


Fig. 5—Front view of Rototrol arc-furnace regulator panel with Optimelt control.



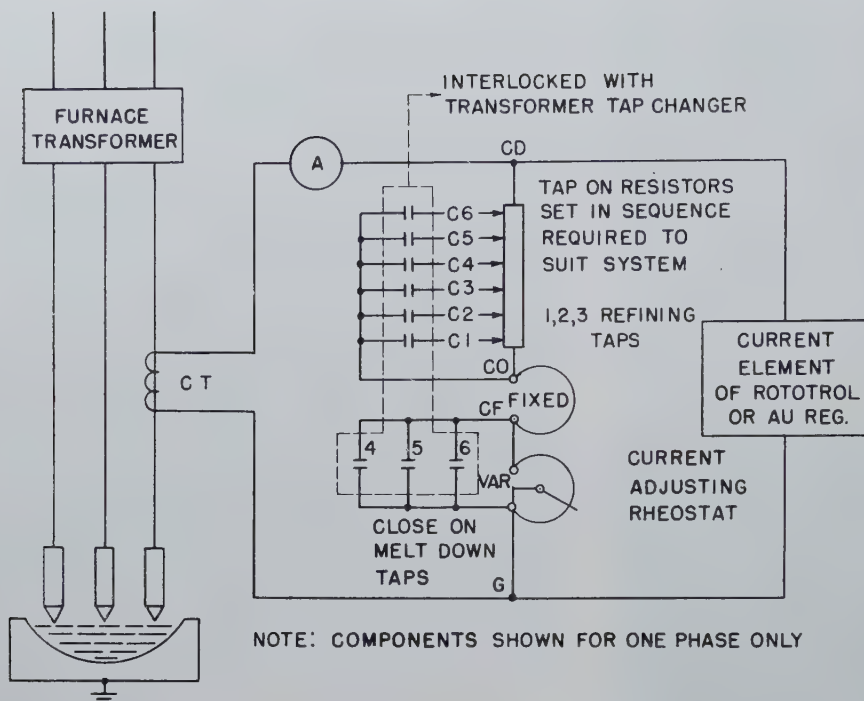


Fig. 6—Simplified schematic diagram of Optimelt control.

**Calibrating Adjustments:** It will be of general interest to record the practical methods which have been discovered for the adjusting of Optimelt control for the desired conditions. Anyone who has observed the operation of an arc furnace will agree that it is far from practical to attempt any direct measurement of actual arc KW. For similar reasons it is also impractical to measure the impedance of the various portions of the secondary circuit. Actual operating conditions are necessary since temperature plays a part in the resistance of many portions of this circuit.

The most practical method of making the Optimelt control resistor settings on an arc furnace is to work from average curves plotted from graphic instrument readings. For the first step a series of simultaneous graphic instrument charts should be taken of the three-phase KW input to the furnace transformer and of each of the three secondary currents. Such a test setup is shown in fig 7. These tests are taken on each of the taps to be controlled. Each individual record of a particular tap voltage should embrace operation at a wide range of current adjusting rheostat positions and operations on each setting should be long enough to catch a few fairly steady points of regulation. From these records points can be plotted for a curve between the secondary currents of each phase and the three-phase input circuit KW. All points are plotted and from this family of points a smooth representative curve for that particular tap voltage and phase is obtained. This curve will be similar to those shown in fig 1. From these curves it will be very apparent where the maximum circuit KW point falls.

In some cases it will be found that the maximum circuit KW cannot be obtained on the highest voltage tap due to limitations in the relations between secondary impedance and the furnace transformer size.

In other words, for some installations the furnace transformer KVA is a bit low for the furnace it operates and although it probably is OK for the service desired, it is still impossible to secure the optimum KW point on the highest secondary voltage. In such cases the final Optimelt control setting on this point would be governed by the permissible maximum KVA loading which the transformer will stand.

Supplementary tests are then taken, with the furnace idle, to establish a relation between secondary regulated current and the actual resistance changes in the current adjusting resistors. The preferred method is to disconnect the ungrounded lead from the secondary current transformer of the phase to be tested. For safety the current transformer secondary should be shorted. An ordinary meter tester's load box operating on the same frequency as the furnace supply is connected between the disconnected regulator current lead and ground as shown in fig 7. Two ammeters are placed in the regulator circuit. The first should be the same graphic ammeter used to obtain the secondary current vs input circuit KW curves described above, and this is, of course, connected to read the entire current to the regulator current element and its shunting resistance. The second ammeter is an indicating instrument and measures the current passing through the current element of the regulator alone. The regulator manufacturer should be consulted and the balancing current for that particular regulator obtained. Now the load box is adjusted to provide a secondary voltage which will circulate this balancing current in ammeter No. 2. The setup is such now as to properly adjust the Optimelt control resistors.

Taking the regulator a phase at a time, the curves for each of the voltage taps of that phase are consulted. For each tap point the secondary current for



the maximum circuit KW point is read off the curve. This current value is reduced 10 pct and used as the approximate point at which maximum arc KW will occur. As explained above, it has been found impractical to measure this value with accuracy. This method of approximating the desired point has yielded very satisfactory results on a number of installations. As explained later, this is the starting point and vernier adjustments based on tabulated monthly efficiency records will permit the selection of the best final adjustments.

The actual current value calculated above is now brought to the test setup shown in fig 7. The tap changer is run to the tap position to be adjusted. The proper Optimelt control resistor tap is then adjusted until the proper secondary current appears at graphic ammeter No. 1. At the same time corrections are made on the load box to maintain the proper regulator current at ammeter No. 2.

These adjustments are continued for each tap to be controlled on the particular phase and then repeated for the other phases.

If control is provided for the refining taps, the maximum permissible secondary current for each tap is recorded. This can best be set by the actual observations of a competent melter. After this value is available, the resistors are adjusted during the shut-down test illustrated in fig 7. The current adjusting rheostat is moved to the maximum current position and the tap changer set on the refining tap being tested. The Optimelt control resistor tap is adjusted to give the desired permissible maximum current and, as before, a check is made to make sure the proper regulator current is maintained.

When the Optimelt control resistor adjustments for all taps and all phases have been completed, the

furnace should be put in regular service. The regular production records should be tabulated for a specific period. Records of KW-hours per ton, tons per hour, hours per heat, and pounds of electrodes per ton are particularly useful for comparison purposes. At the conclusion of this test period move all of the Optimelt control resistor taps for the predominate melting taps a small and equal percentage of the secondary current in the same direction and observe for another duplicate period. Record comparisons will soon establish the final and most efficient point for each tap. It should be noted that the first approximate settings secured from tests will usually yield remarkable savings over the previous hand methods of adjustment. The operator, therefore, is collecting good savings during the extended periods where these refinements in the final settings are being obtained.

**Conclusion:** The Optimelt control system of controlled arc-furnace regulator settings can be expected to yield the following benefits:

- (1) Highest values of power factor corresponding to maximum power input requirements.
- (2) Less KW-hours per ton.
- (3) Less time per heat.
- (4) Positive protection against impairment of product analysis by carbon pickup.
- (5) Improvement in refractory life.
- (6) Reduced consumption of electrodes.
- (7) Consistent duplication of operation from heat to heat.
- (8) Proper utilization of installed electrical equipment.
- (9) Elimination of human error and the time required to make manual adjustments of the electrode current.

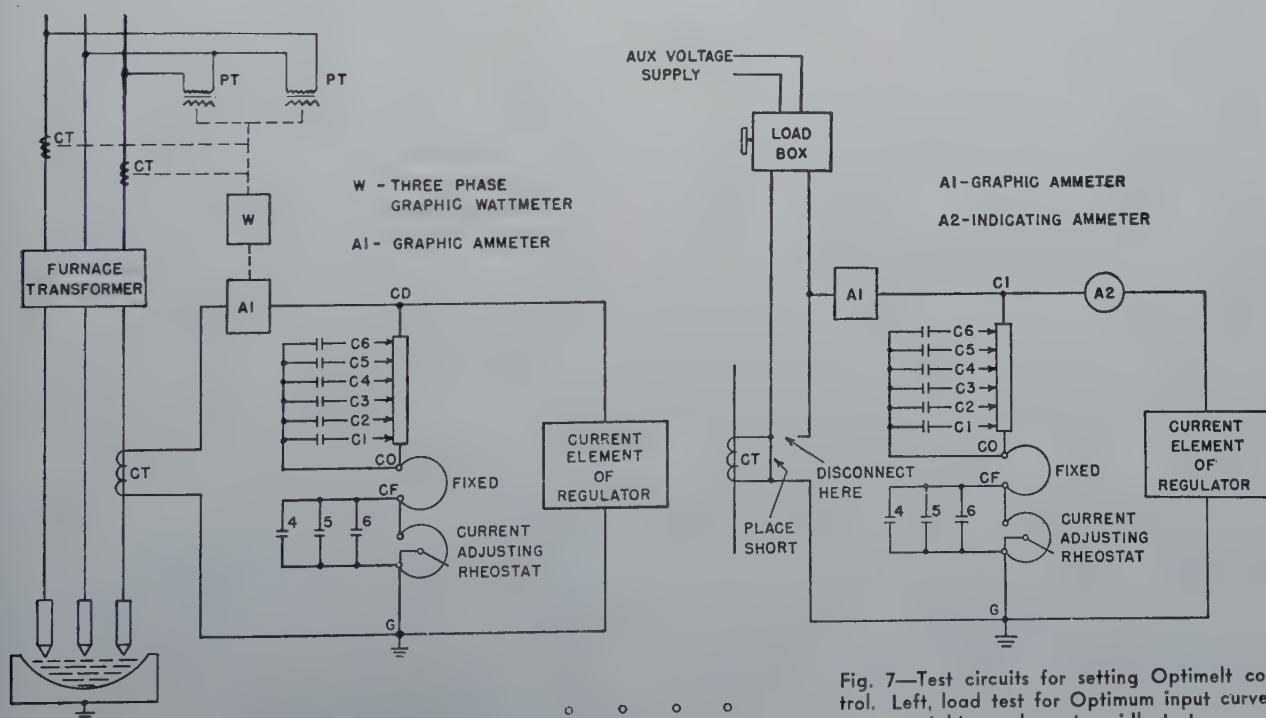


Fig. 7—Test circuits for setting Optimelt control. Left, load test for Optimum input curves; right, supplementary idle test.



# The Bessemer Converter Process

by A. B. Wilder



A TYPICAL acid bessemer converter is shown in fig 5. This type of converter may be used for the production of a 30-ton blow although the rated capacity is 25 tons. After thickness of the lining has been reduced by erosion, considerably more iron may be added and the size of the blow increased. In addition to lining wear in the shell, erosion of the tuyeres also increases capacity of the converter. The mouth of the converter is enlarged by continued use and this not only affects the gas pressure in the converter during the blow, but also changes characteristics of the flame.

The depth of the bath in the bessemer converter influences the nitrogen content of the finished steel. The depth of bath in the converter shown in fig 5 with 25 tons of molten iron is 30 in. with a 16-in. lining and after the lining is reduced to approximately 6 in., the depth of bath is about 20 in. This decrease in depth of bath will slightly reduce the nitrogen content of the blown metal as the contact of nitrogen with the molten metal is less. Further, the iron oxide film of the bubble in a shallow bath may, under favorable conditions, decrease the diffusion rate of nitrogen into the molten metal. Temperature and chemical composition of the bath also influence the behavior of nitrogen.

A shallow bath may also be incorporated in converter design by enlarging the diameter of the shell above the level of the tuyeres. This feature has been described by Brassert<sup>4</sup> and is shown in fig 6. A design of this type increases the capacity of the converter.

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In addition, convection currents of molten metal in the converters are changed, due to the shape of the vessel, and this may result in decreased blowing time.

The shape of a basic converter was investigated by Heyden<sup>5</sup> in Dortmund, Germany. In 1930 the capacity of a 20-ton vessel was increased to 30 tons without changing the converter base or trunnion design. The new converter design is shown in fig 7. The wear on the back was more severe than on the belly and uniform on the sides as shown by the dotted lines. Wear measurements were taken after 308 blows involving 8584 net tons of molten blast furnace iron on five bottoms. In a converter campaign with 32-ton charges, the blowing time on five bottoms decreased from 0.92 min per ton of iron to 0.38 min per ton of iron at the end of the campaign. The volume of blast increased from 9550 cfm for the first bottom to 20,500 cfm on the fifth bottom and depended upon the degree of "slopping." Dolomite consumption was decreased with the new converter design. The depth of bath during the life of each bottom increased with bottom wear. However, for the converter campaign, the average depth of bath appreciably decreased. Bath depth was based on calculations of the space conditions in the lower part of the converter rather than the true depth of bath during the blow.

The results obtained by Heyden indicate the capacity of existing converters in this country may be increased by enlarging the converter shell without changing trunnion design. It may be necessary, in certain converter plants, to provide additional wind capacity and better handling facilities with the oval shaped converter. The economic advantages of larger converter capacity should be evaluated more thoroughly. Changes in converter lines should be made on an experimental basis, as behavior of the molten



**I**N the first half of this two-part article, the author last month reviewed the early history of the pneumatic converter process, investment costs, phosphorus and sulphur limitations, scrap market conditions and deoxidation of various elements. Herein, technological developments are described, as well as oxygen-enriched blast, end-point evaluation and temperature control.

metal during the blow may be adversely affected, resulting in spitting, greater refractory consumption and longer blowing periods. At the Friedrich-Alfred-Hutte<sup>1</sup> plant, Rheinhausen, Germany, bottom-blown converters were designed for 60 to 80 tons capacity with a 1:1.3 ratio of diameter to height instead of 1:1.7 for 20 ton and 1:1.5 for 60 ton converters in operation in Germany.

The nitrogen content of the finished steel is in-

fluenced by the size of tuyere holes. Tuyeres with a  $\frac{7}{8}$ -in. instead of a  $\frac{5}{8}$ -in. size hole but with the same total area of holes, have been employed in a 15-ton converter for the production of steel with 20 pct less nitrogen compared with regular bessemer steel. An optimum increase in tuyere hole size may not adversely affect conditions with respect to "slopping" during the blow provided the proper bottom design with blanked out tuyeres is used. The large size tuyere holes produce a larger gas bubble and, therefore, for the same volume of air compared to the

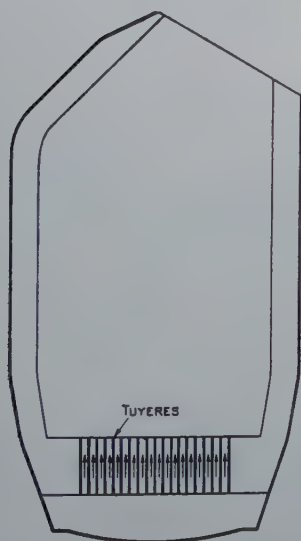


FIG. 5 — (Left) A typical 25-ton acid-bessemer converter.

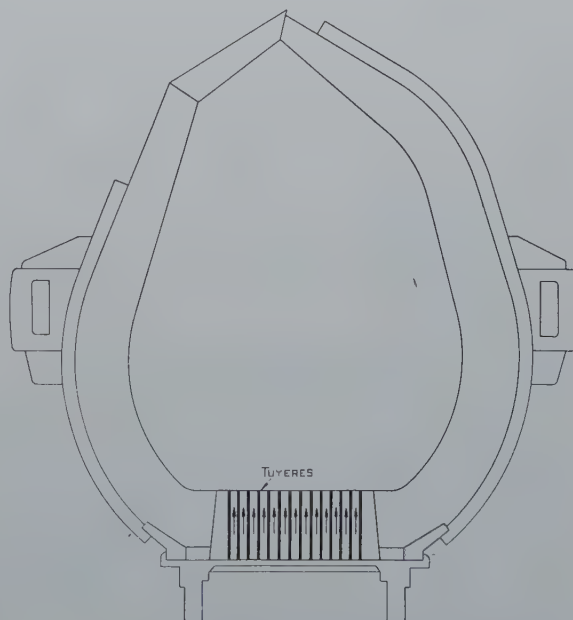


FIG. 6—(Right) A pear-shaped converter.



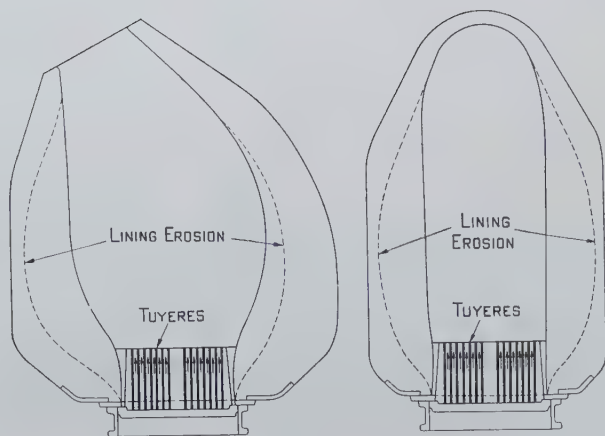


FIG. 7—An oval-shaped converter.

smaller size tuyere holes, the area of contact of the gas bubbles with the molten iron is reduced. The iron-oxide film in the bubble may also be retained for a longer period.

Another feature of converter design is the side-blown converter principle. Small side-blown converters up to 6 tons' capacity have been used in the foundry in the production of steel. A conventional 13.5-ton bottom-blown converter with a modified bottom adaptable to side blowing is shown in fig 8. This converter design was successfully used in the production of 10-ton heats for the manufacture of butt-welded and seamless pipe. The nitrogen content of the finished steel was appreciably reduced by side blowing, as shown in table IV. It will be observed the nitrogen content of the bottom blown bessemer heats is higher than usual. A number of side-blown heats not shown in the table had a nitrogen content of 0.008 pct. Work-brittleness tests<sup>11</sup> of seamless pipe in the as-rolled condition manufactured from side-blown steel deoxidized with molten iron, silicon, and aluminum are shown in fig 9. The results, however, were similar to those obtained with the same type of deoxidation practice in a bottom-blown converter. The similarity of results with aluminum-killed steel in bottom and side-blown vessels was due to the fixation of nitrogen by aluminum. Therefore, in the manufacture of seamless pipe the bottom-blown converter design may be used in the production of steel with mechanical properties similar to open-hearth steel, provided aluminum or its equivalent is properly used for deoxidation. The toughness characteristics of capped, rimmed or silicon-killed steels may be improved by lowering the nitrogen content of the steel.<sup>8</sup>

Operation of the side-blown converter shown in fig 8 was in general similar to bottom-blown practice. The end-point characteristics, however, were somewhat different and the blowing time was increased about 25 pct. This latter factor influences refractory consumption. Ignition of the blow did not present a problem although this may be a factor, depending upon the temperature and composition of the iron. The side-blown process is primarily adaptable to the production of "open" steels for wrought products and killed steels in the foundry.

Side-blown basic-bessemer converter practice developed in Germany has been described by W. Badling.<sup>12</sup> A 30-ton basic converter with a tuyere block inserted in the lining and arranged for immersion and some surface blowing is shown in fig 10. This design may be compared with the 2-ton side-blown acid converter employed in the steel foundries of this country and shown in fig 11. It will be observed the air blast in the foundry converter is directed at an angle toward the surface of the charge resulting in surface blowing. In the German side-blown basic converter, the tuyere block had 90 holes 1.0-in. in diam compared to 220 holes, 0.6-in. in diam, for bottom blowing practice and the tuyeres were immersed in the bath. The dotted lines in fig 10 indicate the type of erosion experienced with the lining. The large-hole tuyeres were employed for side-blowing due to the observations of Naeser<sup>13</sup> which are summarized as follows:

- (1) Excess oxygen must be present in the gas bubble to reduce nitrogen absorption.
- (2) A favorable tuyere diameter must be employed to insure the presence of excess oxygen.

The side-blown basic converter was able to produce 130 blows per lining compared to 250 blows for normal bottom-blown converter practice. The wear in the side-blown converter occurred exclusively in the belly side and was due to high temperature and excess oxygen. The experimental use of special refractories indicated the converter lining life of the side-blown vessel may be extended to 200 blows. The tuyere block life was approximately 25 blows. Due to the fact that the blowing time of a bottom-blown vessel was 0.7 min per ton compared to 1.2 min per ton for side-blowing, the latter type of vessel lining would be expected to last about 150 blows.

Nitrogen content of the side-blown basic-bessemer steel made in the converter shown in fig 10 was very low and is depicted in fig 12. The phosphorus content

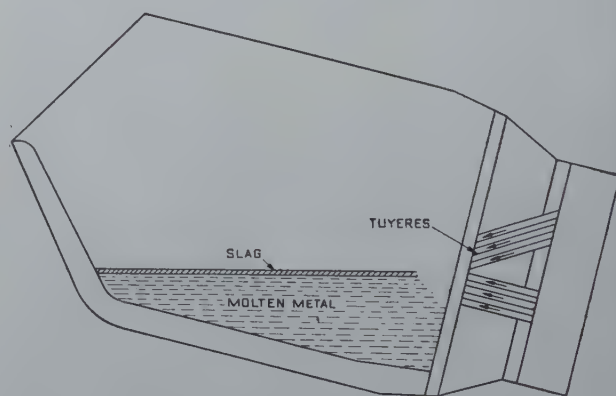


FIG. 8—Side-blown acid converter.

of the various blows was below 0.05 pct. At the Mannesmann Tube Works in Germany, the blooming mill yield of side-blown aluminum-killed converter steel was about 80 pct. Behavior of side-blown steel in the piercing mill was similar to openhearth steel. Many tests with deep drawing and other applications indicated side-blown basic-converter steel to be similar to openhearth steel.



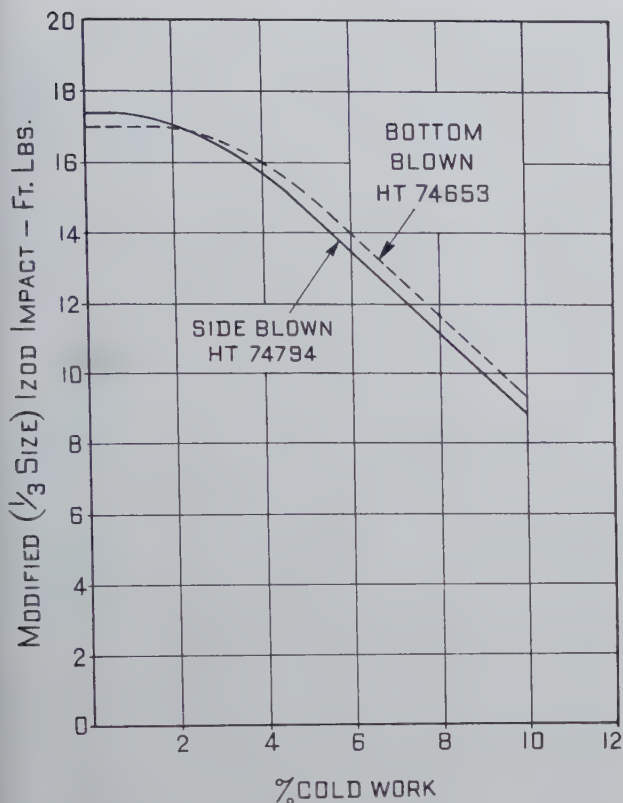


FIG. 9—Embrittlement tests of side-blown converter steel.

A sample of 5/16-in. seamless tubing made in a 35-ton side-blown bessemer converter at the Mannesmann-Rohrenwerk-Huckingen Plant in Duisberg, Germany, and examined in the laboratories of National Tube Co. has the following chemical composition.

C	Mn	P	S	Si	Sl
0.10	0.32	0.090	0.039	0.156	0.054

The steel contained a considerable number of alumina inclusions indicating the blown metal was probably not deoxidized with carbon before treatment with aluminum.

In side-blowing practice the air is usually blown over the surface or through a shallow bath of molten metal. Impurities are principally removed by iron oxide diffusion into the bath and nitrogen in the air has only a limited opportunity to diffuse into the molten metal. In side blowing, the combustion of CO to CO<sub>2</sub> in the vessel above the bath improves the thermal efficiency of the process. Side-blown converter steel in general has a higher temperature, unless proper precautions are taken to control this factor. A thorough evaluation of metal losses in large side-blown converters has not been made and should be considered in a development of this type as metallic yield in small side-blown converters in the steel foundry is lower compared to large bottom-blown converters employed in the production of ingots. A properly designed side-blown converter with a capacity of 25 tons or more should have a metallic yield comparable with bottom-blown practice. The air pressure in a side-blown converter may be as low as 5 psi compared to 30 psi required

in bottom blowing. This is an important economic consideration, but can be realized fully only in a converter plant designed with low pressure turbo-blowers.

Recently, a new approach to the control of nitrogen and phosphorus in steel has been developed in this country.<sup>14</sup> Steel of openhearth quality has been made from pig iron of a composition normally used in the basic openhearth process by refining the mol-

<sup>9</sup> H. A. Brassert, "Bessemer Converter," U. S. Patent 2,265, 511, Dec. 9, 1941.

<sup>10</sup> T. Heyden, "Investigations of the Most Favorable Shape of the Basic Converter," Stahl und Eisen (1925), Vol. 54, pp. 1225, 1256 and 2934.

<sup>11</sup> H. W. Graham and H. K. Work, "A Work Brittleness Test for Steel," Proceedings ASTM (1939), 39, p. 571.

<sup>12</sup> W. Bading, "The Development of Basic Converter Practice," Stahl und Eisen (1947), Vol. 66-67, pp. 137, 180 and 212.

<sup>13</sup> G. Naeser, "Confidential Report No. 67, Part 1," Verin Deutsche Eisenhüttenleute, May, 1944.

<sup>14</sup> The Turbo Hearth, Iron Age (1949), April 21, p. 63.

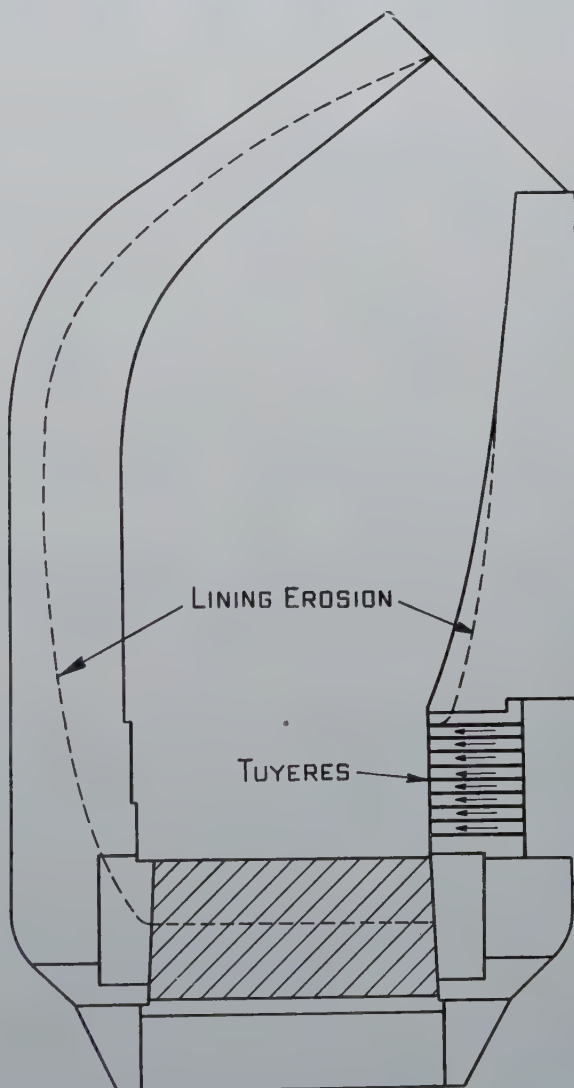
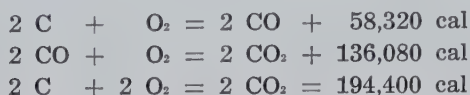


FIG. 10—Side-blown basic converter.



ten iron in a "turbo-hearth" consisting of a vessel with a basic lining and tuyeres arranged for surface blowing. In addition to the use of a basic lining, steel has also been made by this process with an acid lining. In the acid process, phosphorus was not removed but a low-nitrogen steel was produced. Experimental work has been conducted in ½-ton, 3½-ton and 22-ton vessels with a large percentage of the ingots converted to commercial products.

The combustion of carbon to carbon dioxide in the "turbo-hearth" takes place to a large extent inside the vessel. In a bottom-blown converter, carbon monoxide forms inside the vessel and is burned to carbon dioxide in the air outside the vessel. The heats of reaction are as follows:



The formation of carbon dioxide inside the "turbo-hearth" eliminates the necessity for high silicon iron normally required in the bottom blown acid converter or high phosphorus iron in the bottom blown basic converter. The heat of combustion of carbon is, therefore, more fully utilized in the "turbo-hearth," as it is in the side blown converter, and

TABLE IV					
Chemical Composition of Side-Blown and Bottom-Blown Killed Bessemer Seamless Steel					
Blow No.	Side-Blown Bessemer				
	C	Mn	Si	N	Al
30106	0.24	0.52	0.16	0.010	0.055
45788	0.17	0.57	0.19	0.012	0.016
74794	0.17	0.53	0.18	0.011	0.063
	Bottom-Blown Bessemer				
	C	Mn	Si	N	Al
30103	0.19	0.60	0.19	0.016	0.034
45790	0.16	0.52	0.19	0.017	0.035
74653	0.14	0.48	0.17	0.020	0.056

this conservation of heat is the thermal basis for the new process. The metallurgical principles in the "turbo-hearth" process appear to be fundamentally sound and the operating problems should not be insurmountable.

Converter design has been investigated by Kootz and Gille.<sup>15</sup> A small converter of plastic material was constructed in the laboratory. Water, air, ammonia, cork and oil were employed in the experimental work. A stroboscope was used to study bubble action and slopping. The results were of an exploratory nature and in certain instances applicable to commercial practice.

The future design of converters will probably be directed to the construction of vessels with a larger capacity in order to reduce the cost of bessemer steel. A number of large bottom-blown converters with capacities exceeding 50 tons were in operation in Germany during the war. At the present time, the size of a heat may be increased by pouring several bessemer blows in one ladle and this practice has some of the advantages which are achieved in

openhearth practice with larger furnace capacities.

Changes in converter design require thorough evaluation with respect to operating characteristics. The problems encountered in openhearth design are of an entirely different nature and if a similar effort had been directed in this country to converter design and practice, considerably more progress would have resulted. Present trends in the use of oxygen and compressed air in the bath of openhearth furnaces indicate clearly the future possibilities of a more widespread use of the basic principles of the pneumatic converter process.

### Oxygen-Enriched Blast

The first practical investigation of the use of an oxygen-enriched blast was carried out in a bottom-blown basic converter in 1925 and described by A. Bruninghaus<sup>16</sup> and J. Haag.<sup>17</sup> The experiments conducted in Germany indicated the possibilities of increased production and greater use of scrap. The heat balance of a basic converter with oxygen-enriched blast had been previously developed as early as 1908 by F. Wust and L. Leval.<sup>18</sup>

The results obtained in a 10-ton bottom blown basic converter at Rosenberg, Germany, were described in 1939 by W. Eilender and W. Roeser.<sup>19</sup> The blast contained about 30 pct oxygen and the experiment was initially conducted in 1930. The quality of steel and consumption of deoxidizers were not affected. Length of blows and nitrogen content of the steel were decreased. The scrap charge was increased.

A special 1½-ton bottom-blown acid converter was

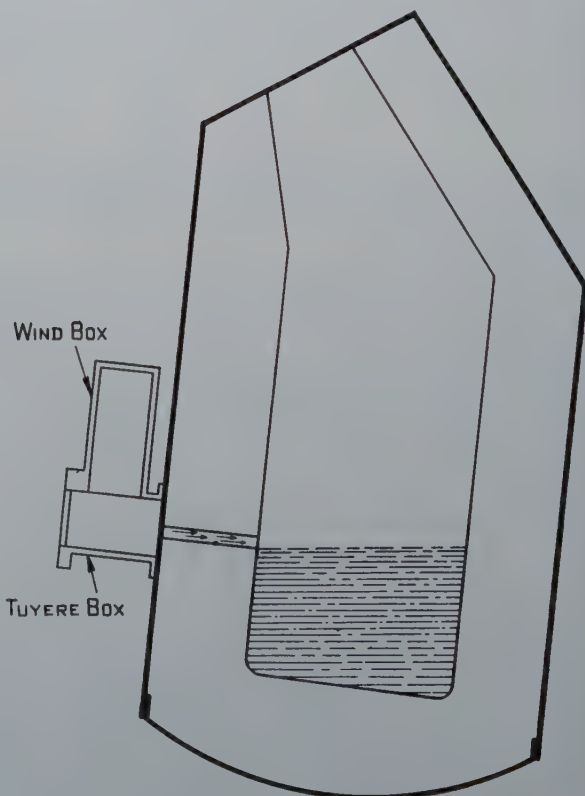


FIG. 11—Side-blown foundry converter.



initially operated at the Kuzentsk Metallurgical Combine in Russia in 1945.<sup>20</sup> Fifteen heats were made with a 50 to 100 pct oxygen blast. Some difficulty was experienced with "slopping" due to a change in shape of the tuyeres which resulted from the high concentration of oxygen used.

The use of oxygen-enriched blast in a 25-ton bottom-blown acid converter has been described by Strassburger.<sup>21</sup> A series of tests were made using about 200 cu ft of oxygen per ton of metal. The oxygen was used for 3 to 4 min during the scrap melting period and 1 to 2 min during the carbon blow for final reduction of silicon. The results are summarized in table V. The blowing time was decreased about 30 pct and the tons of blown metal produced per minute increased approximately 40 pct. Experimental work has been conducted also by the Lorain Works of National Tube Co. with oxygen-enriched blast in a 15-ton bottom-blown acid converter. Ten experimental heats were blown in the same converter with approximately 30 pct oxygen blast and comparison heats with regular converter practice were also blown. The decrease in blowing time with oxygen heats was approximately 3 min and the cold-metal charge was approximately 30 pct for the oxygen-enriched blows and 10 pct for the comparison blows. The tuyere loss during an oxygen-en-

riched blow was approximately 1/2 in. and comparison blows were slightly less. Erosion of the tuyeres was determined by measuring three or four holes of each individual tuyere before and after each oxygen-enriched and comparison blow. Vessel lining erosion was determined by visual inspection before and after each blow. The pouring and teeming temperatures were similar for both types of blows. A typical chemical composition of the metal is shown in table VI and the slag in table VII.

The results obtained with oxygen-enriched blast may be summarized as follows:

- (1) Shorter blowing time.
- (2) Greater utilization of cold iron and scrap.
- (3) With a blast containing about 30 pct oxygen, the bottom and vessel linings were not appreciably affected in a limited number of heats.
- (4) Steel quality was similar to regular bessemer heats.
- (5) Flame characteristics were readily distinguishable by blowers and satisfactory teeming temperatures were obtained.

The cost of oxygen in the experimental work at

TABLE V		
Influence of Oxygen on Converter Operation		
	Without Oxygen	With Oxygen
Hot-metal charge, lb.	45,500	45,917
Scrap	9,000	9,000
Total	54,500	54,917
Blow time, min.	9	6.34
Steaming, min.		1.62
Oxygen used, cu ft.		5,950
Time oxygen, min.		4
Product tons per min.	3.00	4.33

TABLE VI					
Chemical Composition of Converter Steel Blown with Oxygen-Enriched Blast					
	Iron	Blown Metal		Ladle	
		Oxygen	Comparison	Oxygen	Comparison
C	4.40	0.02	0.02	0.09	0.09
Mn	0.49	0.05	0.03	0.38	0.38
P	0.068	0.076	0.077	0.079	0.077
S	0.019	0.037	0.032	0.041	0.031
Si	1.20			0.003	0.007

TABLE VII				
Chemical Composition of Slag				
	Vessel		Ladle	
	Oxygen	Comparison	Oxygen	Comparison
FeO	19.60	20.07	17.80	14.79
Fe <sub>2</sub> O <sub>3</sub>	2.27	1.86	1.57	1.43
SiO <sub>2</sub>	59.40	62.62	40.60	37.60
MnO	11.95	11.10	33.81	41.07
CaO	1.30	1.11	0.88	0.81
P <sub>2</sub> O <sub>5</sub>	0.02	0.01	0.01	0.01

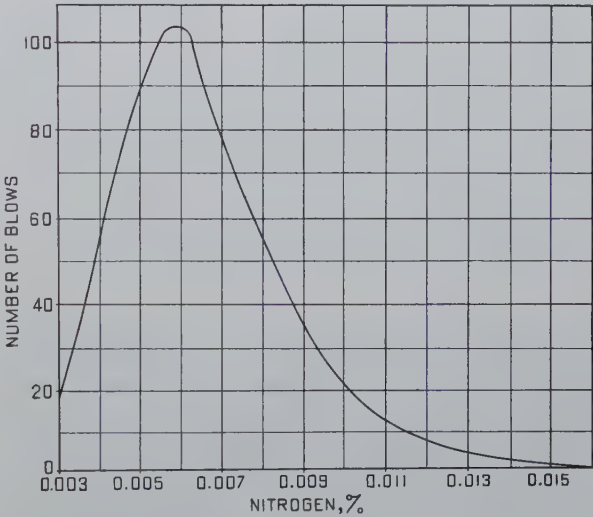


FIG. 12—Nitrogen content of side-blown basic-converter steel.

\$2.62 per 1000 cu ft was approximately \$2.40 per net ton of ingots. An oxygen unit for blast furnace use designed to provide sufficient oxygen for a converter plant would probably provide an economic means for converter operation. In the experimental work, an accurate analysis of metal yield was not obtained due to a limited number of heats. In addition, it would be desirable to evaluate more fully refractory life and the efficiency of ladle additions for a precise economic evaluation of the process.

In the basic-bessemer blow, an appreciable quantity of nitrogen is absorbed in the latter part of the blow as shown in fig 13. Results obtained in a basic-bessemer blow in fig 13 from Dardel<sup>22</sup> were chosen to show the rapid rate of nitrogen absorption



after the drop of the carbon flame. Nitrogen absorption, after the removal of carbon is dependent upon time and temperature. At the beginning of the refining period, the amount of nitrogen in the metal changes very slowly. If the nitrogen content, however, is high, it will be reduced; if low, increased. In the acid-bessemer process used in America, blowing is stopped at the drop of the carbon flame resulting in a lower nitrogen content in the finished steel.

Reduced blowing time and increased use of scrap are important economic factors which favor the use

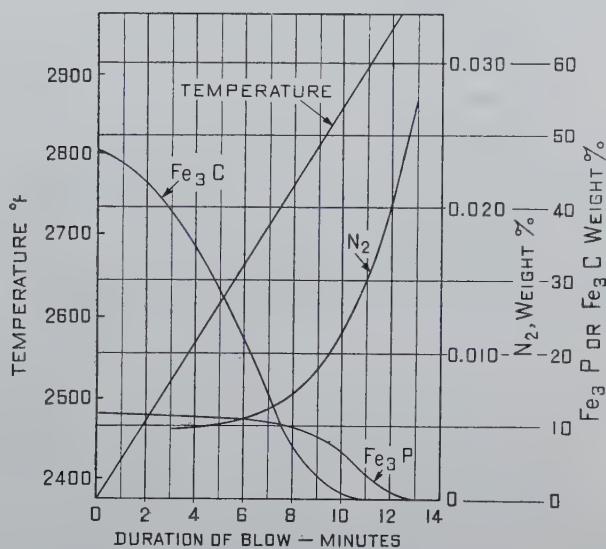


FIG. 13—Typical bath analysis during a basic blow.

of an oxygen-enriched blast. A proper balance of these factors, particularly in periods when scrap is plentiful, will be of considerable importance in reducing the cost of converter steel. One of the outstanding advantages of oxygen-enriched blast is the increased use of scrap and this is supported by heat balance studies of the U. S. Steel Corp. Research Laboratory.

It may also be possible to blow low-phosphorus iron in basic converters with oxygen-enriched blast. This practice would provide additional heat which should be considered in the basic process. Basic-bessemer iron in Europe contains over 1.5 pct phosphorus and by using basic-bessemer slag in the blast-furnace charge the phosphorus content may be maintained at a satisfactory level. In America, during 1947 the low-phosphorus ores for basic iron averaged 0.072 pct phosphorus and the high phosphorus ores 0.408 pct P. Most of the basic iron contains less than 0.25 pct phosphorus; and if a basic converter with oxygen-enriched blast produced steel with a phosphorus content similar to basic openhearth practice, certain economic advantages may be gained.

#### End Point Evaluation

The spectroscope was one of the first instruments used to control the bessemer blow. Roscoe, as early

as 1862, used a spectroscope and other early investigators included Watts and Lislegg in 1867, Kohn in 1868, Drown in 1871 and Greener in 1874. About 1900 Hartley discussed the use of the spectroscope. Kelly<sup>23</sup> in his early attempts to control the end point of the converter blow, determined the malleability of spittings from the vessel with a hammer and anvil. Hartschorne<sup>24</sup> in 1891, regulated the quantity of air blown into the converter after the end point was determined by the spectroscope. Burgess<sup>25</sup> in 1917, used an optical pyrometer, and Fierich<sup>26</sup> in 1928 employed a radiation pyrometer.

<sup>15</sup> T. Kootz and G. Gille, "Investigation of Converter Shapes and Blowing Conditions for the Production of Low Nitrogen Steel," *Stahl und Eisen* (1948) Vol. 68, No. 17/18, Aug. 12.

<sup>16</sup> A. Bruninghaus, *Stahl und Eisen* (1925) Vol. 45, p. 737.

<sup>17</sup> J. Haag, "Use of Oxygen-enriched Blast in the Basic Bessemer Process," *Stahl und Eisen* (1925) Vol. 45, p. 1873.

<sup>18</sup> F. Wust and L. Laval, *Metallurgia* (1908) Vol. 5, p. 431.

<sup>19</sup> W. Eilender and W. Roeser, "Metallurgical Investigations of the Workings of the Basic Bessemer Process with Oxygen-enriched Blast," *Stahl und Eisen* (1939) Vol. 59, p. 1057.

<sup>20</sup> V. V. Kindakov and A. A. Baikov, "Experimental Melting of Steel with Oxygen Blast by Acid Bessemer Process," *Bulletin De L'Academie Des Sciences de Leurss, Classi des Sci. Tech* (1946) No. 10, p. 1401.

<sup>21</sup> J. H. Strassburger, "Tonnage Oxygen for Increased Iron and Steel Production," *Yearbook of the American Iron and Steel Institute* (1945) p. 214.

<sup>22</sup> Y. Dardel, "Manufacture of Basic Bessemer Steel Low in Nitrogen," *Metal Progress* (1947) Vol. 52, No. 2, p. 254.

<sup>23</sup> J. N. Broucher, "William Kelly: A True History of the So-called Bessemer Process" (1924) p. 83.

<sup>24</sup> J. Hartschorne, "The Basic Bessemer Steel Plant of the Pottstown Iron Co.," *Trans. AIME* (1892) Vol. 21, p. 743.

<sup>25</sup> G. K. Burgess, "Temperature Measurements in Bessemer and Open Hearth Practice," *Trans. AIME* (1917), Vol. 56, p. 432.

<sup>26</sup> R. Fierich, *Stahl und Eisen* (1938) Vol. 48, p. 1233.

<sup>27</sup> S. T. Jazwinski, "The Control of Tropenas Converter Blowing by a Direct Vision Spectroscope," *Journal of the Iron and Steel Institute* (1945) Vol. 152, p. 9.

The visual characteristics of the flame continue to

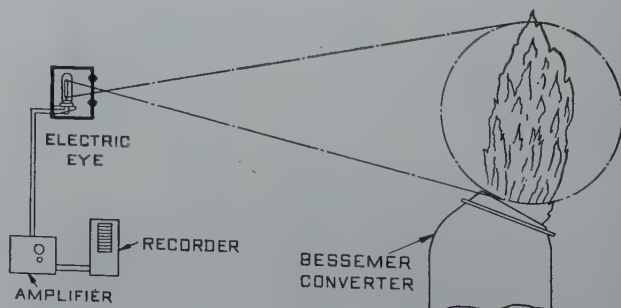


FIG. 14—Photocell arrangement for bessemer flame control.

be the most widely used means for controlling the bessemer blow and determining end point character-



istics. Although the spectroscope has been extensively studied over a period of years as a means for end-point control, this instrument has been used successfully only recently. Marked changes occur in the spectrum at the "end point" of the blow and under suitable conditions may be used as an indicator for turning down the bottom-blown acid converter. It is necessary, depending upon the type of steel being produced, to select a time interval after changes in the spectrum are observed, in order to turn down the converter at the proper carbon content. The amount of natural light and other conditions in the Bessemer plants may interfere with the proper use of the spectroscope and have, therefore, limited its application. Recently, the use of a spectroscope has been described by Jazwinski<sup>27</sup> for determining the end point of a side-blown acid converter. The end of the blow was reached when the red, green, and occasional blue band entirely disappeared.

The use of a photocell for end-point control has recently been described by Work.<sup>28</sup> Arrangement of the equipment for viewing the flame is shown in fig 14. As the flame drops at the end of the blow an arrest occurs which is referred to as the end point.

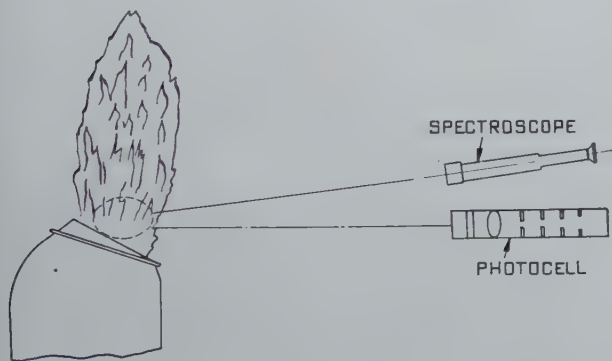


FIG. 15—Arrangement for bessemer flame control.

The number of seconds from the end point until the blow is turned down is called the "afterblow." Each type of steel requires a specific afterblow which is determined by statistical studies of variations in yield and quality in time of afterblow.

Another method for determining the end point of the bessemer blow has been described by Percy<sup>20</sup> of the U. S. Steel Corp. Research Laboratory. The use of a spectroscope and a photocell photometer are described. The radiation measured was restricted to that directly emanating from the converter flame, and the arrangement is shown in fig 15. The blow was controlled to adjust the radiation to follow a predetermined pattern of measurements until the end point was reached.

The use of a spectroscope or photocell for end-point control has found limited application due to variable conditions in various operating units. As technical progress continues in the study of end point control, it may be possible in the not too dis-

tant future, to develop new and improved techniques which will provide a wider application in the use of instrumentation to replace visual observation of converter flame characteristics. One of the most important features of end-point control is the elimination of overblowing. The over blowing of steel results in the absorption of excessive amount of oxygen, fig 3, and nitrogen, fig 13, in the molten steel. This not only affects the efficiency of deoxidation, but also influences behavior of the product during processing in the rolling mills. However, as Graham<sup>30</sup> has pointed out, real progress in bessemer operation will require better control not only of the "end point" but a number of other factors which influence quality and uniform reproducibility of the product. The control of iron quality, for example, leaves much to be desired.

### Temperature Control

The production of steel by the converter process requires careful temperature control in order to insure satisfactory practice. If the finished steel is too low in temperature, ladle skulling and pouring nozzle difficulties will be encountered which are undesirable from the viewpoint of steel quality. Steel with a high temperature not only contains excessive quantities of oxygen, but is also unsatisfactory in other respects. The bessemer blower is therefore confronted with the problem of producing steel at a satisfactory temperature which can only be conveniently determined at the present time with an optical pyrometer, as the blown metal is poured into the ladle or during teeming. Many attempts have been made to develop a satisfactory device to measure temperature of the molten charge during the bessemer blow, but a satisfactory technique has not been

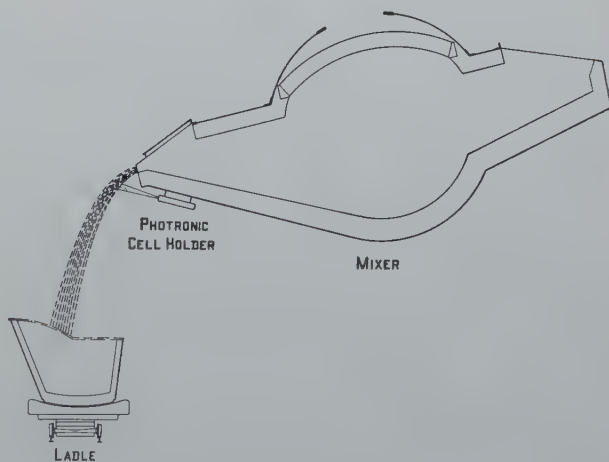


FIG. 16—Arrangement for measurement of mixer-iron temperature.

developed. The blower must continue to depend upon the characteristics of the flame.

Experimental work has been conducted in measuring the temperature of mixer iron with a radiation



device, but a satisfactory procedure has not been developed. A water-cooled radiation head was focused on the stream of molten metal below the pouring lip of the mixer and a recorder installed in the bessemer pulpit. Air was blown through the tube. This is schematically shown in fig 16. One objection to a device of this nature is that changes in the cold-metal charge of the converter may only be made after molten iron is poured in the transfer ladle. However, as a general guide to the blower, an instrument of this type would be useful providing accurate readings were obtained. The accuracy of readings depends essentially on atmospheric conditions at the mixer and the nature of the stream of molten iron.

Chemical composition of the iron is a fundamental consideration with respect to temperature control, particularly with regard to silicon. The oxidation of silicon is an exothermic chemical reaction and information as to the silicon content of the iron should be available to the blower. If the silicon content is high, cold metal or iron ore may be added to the vessel to eliminate the necessity for steaming. If the silicon content is too low, it may be necessary to side blow in order to increase the temperature of the metal. During the blow if the temperature is excessive, all of the silicon may not be removed before carbon oxidation predominates.

A cold ladle or vessel and the amount of ferro-alloy addition at the end of the blow are among the factors which must be considered by the blower in producing steel with a satisfactory temperature. The blower, therefore, must take into consideration the many variables which affect temperature and by judging character of the flame, produce a satisfactory quality steel. The accurate determination of

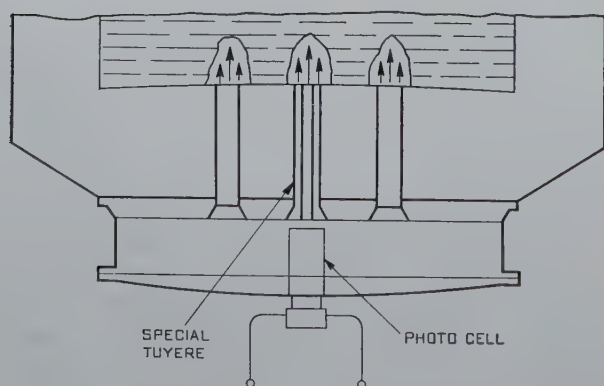


FIG. 17—Arrangement for measurement of metal temperatures during the blow (schematic).

temperature during the blow with a radiation instrument would, therefore, be of considerable significance in the economic production of Bessemer steel.

A photronic cell located in the wind box has been described by J. W. Percy<sup>21</sup> of the U. S. Steel Corp. Research Laboratory and a schematic drawing is shown in fig 17. The possibilities of securing ac-

curate temperature determinations by this method are promising, but additional experimental work will be required to develop a satisfactory technique. Temperature evaluation during the bessemer blow by measuring radiation characteristics of the flame may be compared to the technique employed by the blower, but the application of these principles has been largely confined to end-point evaluation.

### Summary

The history and economic aspects of steel production in the pneumatic converter have been briefly discussed. Certain technical developments with reference to steel production have been reviewed and

<sup>28</sup> H. K. Work, "Photocell Control for Bessemer Steelmaking," *Trans. AIME* (1941) Vol. 145, p. 132.

<sup>29</sup> J. W. Percy, "Bessemer Converter Blow Control Method," U. S. Patent 2,354,400 July 25, 1944.

<sup>30</sup> H. W. Graham, "The Acid Bessemer Process in 1940," *Trans. AIME* (1941) Vol. 145, p. 113.

<sup>31</sup> J. W. Percy, "Bessemer Converter Bath Measuring," U. S. Patent 2,305,442, December 15, 1942.

<sup>32</sup> E. C. Wright, "Manufacture and Properties of Killed Bessemer Steel," *Trans. AIME* (1944), Vol. 158, p. 107.

described with particular emphasis directed toward the future. The pneumatic process appears to be inherently adaptable to the economic production of steel suitable for most of the applications which utilize openhearth steel.

The bessemer process must be more fully developed upon a technological basis to win back the position it once held. Additional experimental work is necessary before acceptable changes in converter design and methods of operation are more fully recognized. The cause of bessemer steel cannot be effectively advanced with steel-making practices which were developed many years ago. Engineering materials of the present day must meet more exacting requirements and at reduced costs. The future economy of the steel industry in terms of correct balance of processes and the security of our nation are, to a degree, dependent upon increased production and wider acceptance of bessemer steel.

### Acknowledgment

The author wishes to express his appreciation for the assistance received from the management and operating personnel of National Tube Co. and the U. S. Steel Corp. Research Laboratory. He is particularly indebted to Messrs. E. G. Price, Vice-President in Charge of Operations; A. W. Thornton, Division Superintendent, Blast Furnaces, Steel Works and Rolling Mills; and W. B. Kennedy, Chief Metallurgist, National Works; W. T. Miller, Superintendent of Bessemer; and J. O. Light, Chief Metallurgist, Lorain Works, National Tube Co., and E. C. Wright<sup>32</sup> for his pioneer work in development of the deoxidized acid bessemer process for the manufacture of seamless pipe.



# Can Leaching Aid the Copper Mines?

THE only living member of the original Tainton technical staff that operated the Martinez electrolytic-zinc plant and proved the commercial soundness of high acid-high current density deposition of zinc tells of the tribulations encountered in that early work. It is then shown that copper leaching may well have a great deal in common with zinc hydrometallurgy and that several proven metallurgical processes could be integrated into complete operations adaptable to a wide variety of copper mine conditions.

by HARMON E. KEYES

ABOUT the year 1913 an ambitious young engineering student at the University of Manchester approached his chemistry professor with a request to experiment in the laboratory on electrodeposition of zinc at extremely high current density and acid strength. The professor explained to the apparently misguided young man that acid dissolved zinc and any further improvements in electrolytic zinc would be with lower rather than higher acidity.

The student, however, had an inquisitive and individualistic turn of mind. He later admitted that just because he didn't know any better, he conducted his high-acidity deposition of zinc with high current density. The result was a dense, adherent coating at high current efficiency, which was a distinct improvement over the standard practice of the day. From that time on he received the cooperation of his professor and full use of available facilities.

Such was the birth of the Tainton high acid-high current density electrolytic zinc process, now applied successfully in leaching zinc ore and electrogalvanizing of steel wire.

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**H. E. Keyes is a Chemical and Metallurgical Engineer, Phoenix, Ariz., and a member of AIME.**

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In the years accompanying Tainton's commercial development of his discovery, the going was tough. Experts around the world attacked his process because it did not fit accepted theory. Thereupon Tainton, with his characteristic resourcefulness, invented a theory to fit the facts. This dealt with the postulation that hydrogen overvoltage increased with current density, thus permitting zinc deposition in preference to hydrogen evolution under environmental conditions conducive to resolution of zinc at the low current densities then in vogue.

Naturally, many practicalities in this revolutionary process had to be solved, and the problem gradually assumed the form of rather involved chemical engineering technology. Those managements that fostered the chemical engineering approach in solving these problems have been

rewarded by successful developments by their engineering staffs.

The writer, being the only living member of the original Tainton technical staff that operated the Martinez, Calif., electrolytic-zinc plant in 1920-21, welcomes the opportunity to record some hitherto unpublished information dealing with typical tribulations encountered in process development work. A bit of the human element during those trying years may be of general interest.

Tainton had made a third futile attempt to secure backing for his project. While enroute from the East he stopped over for some relaxation. In the meantime, funds ran out at the plant. The superintendent, Dan Bosqui, showed a remarkable combination of wit, forbearance and pathos. He gathered wild flowers from the hill and placed them around a slab of zinc set up as a headstone on Tainton's desk, with the following epitaph—

**"Here lie the Merry Zinc Kids  
Asleep beneath the sod,  
Though three times resurrected  
They're dead ones now—by God."**

But the Zinc Kids carried on with spirit and tenacity. Tainton soon arranged a deal with Bunker Hill & Sullivan, and staked the writer with a retainer to continue research in order to establish the technical soundness of electrolytic relationships with high acidity and current density for the deposition of zinc. Tainton had already set up an approach by directing the writer's experiments at Martinez in which, at critical current densities, zinc could be deposited at high acidity on cathodes composed of the very elements that generally wrecked electro-deposition of zinc when present as impurities in electrolytes. This was continued at the physical chemistry laboratory of the University of Washington. Here, actual overvoltage measurements established that the potential for zinc plating could be achieved on cathode surfaces which generally had inhibited zinc deposition. It also was established that the so-called "direct" method of overvoltage measurement was correct, and the previously employed "indirect" or "commutator" method was in error as applied to these conditions.

This piece of research was bitterly attacked on the other side of the globe because it disproved



comprehensive and previously published overvoltage data. However, the method was conceded by Dr. D. A. MacInnes, of M. I. T., to settle a disputed question among scientists, and Dr. MacInnes generously withdrew his own paper in favor of publishing this work of Tartar and Keyes in the *Journal of the American Chemical Society*, March 1922. The promised attack in the technical press from a university in South Africa never materialized. However, during the last War the writer did see in a British technical book, reference to the above research bearing out the established soundness of Tainton's principle.

Tainton's process was ultimately accepted by the Bunker Hill & Sullivan interests for operations at Kellogg, Idaho. From this point on, including his application in electrogalvanizing, the story is too well known to warrant repetition.

It is not the purpose of this article to draw comparisons between the high and low current density electrolytic-zinc processes. The writer was employed at the Trail zinc plant during World War I, when this zinc plant was a "war baby" and it was a struggle to maintain over 30 pct extraction of zinc. This also was a pioneering job, and one that achieved a high degree of metallurgical perfection through constant direction of management in applying chemistry along engineering and practical lines.

A comparison may be appropriate to the field of copper hydrometallurgy. Aside from the fact that copper ores are generally of lower grade and require larger scale mining and mechanical engineering developments, the basic approach to copper leaching may have something in common with zinc hydrometallurgy.

The U. S. Bureau of Mines was responsible for pioneering the "autoxidation" process for use in leaching mixed oxide-sulphide copper ores with dilute ferric sulphate and sulphuric acid. This process provided for simultaneous oxidation of ferrous sulphate and sulphur dioxide, using finely divided air. Many of its successful features involved a reversal of previously accepted concepts. For example, an early type of conversion cell for conducting the reaction consisted of a tank with porous blanket through which dirty  $\text{SO}_2$  gas was blown. Instead of seriously clogging the single-ply blanket, the dust proved beneficial in that more uniform air dispersion resulted from differential arrangement of the dust particles around variable size pores in the blanket. At different times the writer heard recognized metallurgical authorities question the soundness of this process on grounds that "sulphur dioxide is a reducer", and without due cognizance of the physical chemistry governing the practical control of the reaction.

For a period of years the writer has had occasion to make many presentations for leaching at copper mines. A guiding principle in open-drainage leaching is control of solvent according to analysis of the pregnant solution. Two contrasting experiences illustrate this point. An autoxidation plant was built at a dump having a pyrite gangue, the dump itself being a virtual acid plant. Prior to the autoxidation cell design, the management was

quoted as indicating that it "did not want a chemical plant." As the copper mineral was chiefly chalcopyrite, a study of natural acidity, together with the limitations of leaching rate, should have first been made. Later studies raised the question as to whether this added acidic ferric sulphate accomplished any useful purpose in the above instance.

In another situation, the writer made small scale leaching tests, using acidic ferric sulphate. Encouraging results were obtained on a dump material rather high in gangue consumption of acid, and lacking in sulphide constituents adequate to accomplish leaching with water alone. This is an example of logical application of autoxidation to supplant oxidizable sulphides in the ore.

Another example showing need of a better chemical engineering approach to copper leaching occurred while the writer was engaged as a partner in milling a copper ore dump. Preliminary examination showed the copper mineral to be chalcopyrite, and the gangue largely a mixture of ferrous carbonate and talc. Rain water stood on top of the dump until it evaporated. Acid was immediately consumed by gangue, and the coarse chalcopyrite was resistant to ferric sulphate action, even if acidic ferric sulphate could exist in the dump, which was next to impossible. Obviously, leaching was ruled out. One day while the writer was absent on a leaching job, a noted mining engineer visited this milling operation, looked over the dumps and suggested leaching to the other partner as the logical procedure! As a matter of fact, both partners had recently been engaged in a dump leaching project using acidic ferric sulphate, and they would have here followed similar procedure had such been indicated by the chemical relationships involved.

Eleven years ago the writer approached the City of Phoenix with a proposal to develop autoxidation as a cheaper source of ferric sulphate. Due to environmental conditions around a city sanitation plant, a high standard of  $\text{SO}_2$  conversion efficiency was required. Also, a minimum of free acid production was a prerequisite in ferric sulphate intended for coagulating purposes. The problem was solved by design of a new type of  $\text{SO}_2$



Autoxidation ferric-sulphate reactor cells are here combining ferrous sulphate, sulphur dioxide and atmospheric oxygen. This design features chemical stability and mechanical simplicity rather than "flash" reaction. Scrap iron dissolving tank in operation, at lower right.



fixation cell which took advantage of the relatively high solubility of sulphur dioxide and very low oxygen solubility. This design also provided for application to dirty, wet, waste  $\text{SO}_2$  gases by virtue of being able to operate on low pressure of  $\text{SO}_2$  introduction such as obtainable with single-stage centrifugal blowers. A further adaptation, of possible future importance, is in treating steel plant pickle liquor to either produce ferric sulphate or to regenerate pickling strength acid.

The crux of this successful Phoenix development was a critical decision made by Dario Travaini, superintendent of the sewage treatment plant. During a trying period, the City of Phoenix, virtually without funds for research and development, tackled a problem that had just baffled the copper industry. Early in the experimental phase Travaini



Lowered chemical cost is anticipated in this new 30 million-gal-per-day water filtration plant for Phoenix (above), by using autoxidation process. The ferric-sulphate storage solution tank is in the foreground, and the sulphur burner and chemical reaction tanks are at the left.

Partially completed autoxidation ferric-sulphate plant (right). The air blower, motor and sulphur burner are at left center, water plant intake gates are in the left background and the shallow tanks for dissolving scrap iron are in the center. Ferric-sulphate reactor cells are back of scrap-iron tanks.



plainly stated that if the process worked he would put it in, otherwise, he would "kick the — — thing out." The adjectives used are too well understood in western mining parlance to need further explanation. Suffice it to state that the process was installed, and operated successfully during the War years to produce ferrous sulphate for odor control and without special aid of a trained chemist. All this notwithstanding the withdrawal of expected federal funds when the form of aid was changed from PWA to WPA. Later, this batch plant was supplanted by a continuous flow unit of three times the former capacity. This continuous unit has operated successfully for the past two years and produces on full plant scale ferrous sulphate for odor control. Ferric-sulphate for coagulation was produced during plant construction.

A similar ferric-sulphate plant, of six tons rated daily capacity, is now under construction for use in supplying coagulant for the new 30 million-gal-per-day water filtration plant being operated by the City of Phoenix.

Consider now the situation in the steel industry. Steel might well be regarded as the big brother of copper, from a comparison of large scale mining and furnacing methods. Steel seems to have reached almost the ultimate in mechanization in ore reduction and processing, and often has been used as the example of achievement to which copper producers aspire. It is noteworthy that R. E. Zimmerman, Vice President of Research and Technology, U. S. Steel Corp. of Delaware, has shown by examples that chemical engineering concepts are well diffused throughout the steel industry. He also explains what it takes to be a chemical engineer in a metallurgical plant.

**"The Chemical Engineering Concept in the Steel Industry," by R. E. Zimmerman, Chemical Engineering, May, 1946.**

The writer holds the belief that developments in zinc and steel illustrate that improvements in low cost copper production may still be effected in certain types of situations, by application of proven chemical engineering methods; that problems now confronting the copper industry cannot all be solved by dynamite, manpower, construction materials, or even politics; and that the extent to which these copper hydrometallurgical improvements may be adopted advantageously rests large-

ly with decisions of management and receptiveness to the chemical engineering approach.

It has been proven that acidic copper leaching solvent can be produced economically, that cement copper may be recovered effectively by flotation, and that these metallurgical steps may be integrated into complete processes adaptable to a wide variety of situations. Applicability of such methods to specific copper mine conditions is primarily a chemical engineering function, and success of such enterprise depends largely on extent to which this approach is supported by local managements.

Investigation of leaching as possible aid to copper mining poses a thought given in tribute to the late Dr. H. Foster Bain, who was an inspiration to the writer over many years. A bit of advice to enterprising engineer-inventors was printed in the *Mining and Scientific Press* more than 30 yr ago—

**"Never hesitate to make a mistake, but while you are in the mistake-making stage, keep your mistakes on a small scale."**



## Section Activities

(Continued from p. 9)

acted as host for the meeting which included a joint luncheon following the technical session in the morning and a fellowship banquet following the technical session in the afternoon. The banquet was highlighted by formally presenting an oil painting depicting the tapping of a 175-ton openhearth furnace to A. H. Sommer who has been the Section Chairman for the past seven years.

In discussing openhearth combustion and related problems at the morning session, the use of various instruments to check openhearth combustion and efficiency were discussed at considerable length. The determination of both oxygen and combustibles in the waste gases was agreed to be of extreme importance and equipment used for this purpose was described in detail. It was explained that through the use of this equipment, fuel consumption had been reduced as much as 18 pct in one plant.

### Connecticut Section

T. H. Carlson reports that the first meeting of the Connecticut Section of the fall and winter season was held in the University Club in Bridgeport, on Wednesday, Oct. 12. Lewis E. Thelin, of Scovill Mfg. Co., the Section Chairman, presided.

In his opening remarks, Mr. Thelin stressed the desirability of advertising our Section meetings to those who might be eligible as prospective members. The cooperation of the present members was requested in submitting names of such prospects for inclusion in a special mailing list.

Attention was also directed to the Women's Auxiliary of the Connecticut Section which now has about 60 members. Membership is open to wives of present Section members. Members were asked to support the Auxiliary in their efforts to increase their membership. A joint social function is being planned for the future months.

The featured speaker was Dr. Arthur Phillips, professor of metallurgy at Yale University and a past chairman of the Institute of Metals, who spoke on the topic "Metallurgy in Brazil." Mr. Lloyd E. Raymond of Singer Mfg. Co., vice-chairman of the Section and head of the Programs Committee, introduced the speaker.

Dr. Phillips visited Brazil in 1944 and lectured in the Escola Politecnica of the University of Sao Paulo and in industrial centers of Brazil under the joint sponsorship of the United States Coordinator of Inter-American Affairs and the Brazilian Government.

As a preface to his observations on the metallurgical

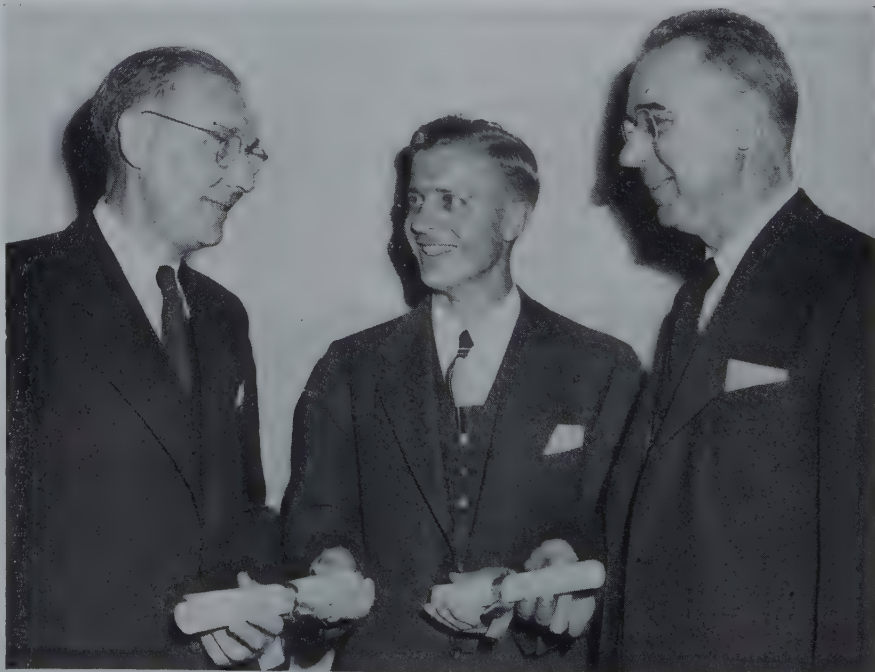
and industrial development of the country, Dr. Phillips traced in detail the political and economic history from the early days.

Most important of the Brazilian reserves is iron ore, constituting 22 pct of the world reserves. The ore is high grade and low in phosphorus and silica but high production rates are prevented by poor location and transportation. Also included in the resources are deposits of manganese ore, nickel ore, bauxite, beryl, tantalite, rutile, monazite sand, quartz crystals having piezo-electric properties, mica and magnesite.

The production of ferrous products is most important in Brazil's metallurgical picture, the greatest stimulus being from the new Volta Redonda plant which is a fully integrated unit. This plant is rapidly filling the gap between the former production capacity of the country and the greater consumer demand. This very excellent mill, costing over \$100 million, was constructed and put into operation under the guidance of U. S. technicians.

In 1945, the production of primary aluminum was started and a second producing unit is now under construction. There have been some minor attempts at lead and tin recovery but there are no significant deposits of copper, lead, tin or zinc ores. With the exception of aluminum and steel, the production of primary metals is practically nil.

It was Dr. Phillips' opinion that the future of Brazil's mineral economy lay in the utilization and exploitation of the less common minerals and ores such as tungsten concentrates and tantalite which are produced in relatively low tonnages but are high in monetary values. The talk was supplemented by illustrated slides presented with the assistance of Dr. W. R. Hibbard, Jr., of Yale University.



A. W. Thornton, Chairman Pittsburgh Sect. of the National Open Hearth Committee, AIME, presenting award certificates to G. B. Luerssen (left) and C. B. Post (right), both of the Carpenter Steel Co., for the paper entitled, "The Interaction of Liquid Steel with Ladle Refractories," which appeared in the January issue of the Journal of Metals.



## ECPD Holds Annual Meeting

Engineers' Council for Professional Development held its annual meeting this year in Chicago, with a record attendance of about 150, not including a sprinkling of ladies. The Council is a joint body of the four Founder Engineering Societies (AIME, ASME, ASCE, AIEE) and the American Institute of Chemical Engineers, the American Society for Engineering Education, the National Council of State Boards of Engineering Examiners, and the Engineering Institute of Canada. Headquarters were at the Edgewater Beach Hotel, and sessions were held on Friday afternoon, Oct. 28, and Saturday morning, with adjournment following shortly after the luncheon on Saturday. Chairman Parker made his annual report at the dinner on Friday, followed by a talk by Robert E. Wilson on "The Attitude of Management Toward Research." Friday morning was devoted to an executive session of the Council at which accrediting was discussed.

Inspection and accrediting of engineering curricula continues the most notable achievement of ECPD. During the past year, delegatory committees visited 74 colleges and universities, with 64 inspections of new curricula and 250 reinspections of curricula previously accredited. Only 39 schools remain to be inspected in the immediate postwar program, according to the report of H. T. Heald, chairman of the Committee on Engineering Schools. Newly accredited engineering curricula in the field of the AIME were: University of Alabama, metallurgical; Colorado School of Mines, petroleum (production and refining); Illinois Institute of Technology, metallurgical; University of Kansas, petroleum; University of Pennsylvania, metallurgical; Princeton University, geological; Rutgers University, ceramic; A&M College of Texas, geological.

Further additions have also been made to the accreditation of curricula of the "technical institute" type, curricula in seventeen such institutions now having been approved.

Ole Singstad, chairman of the Committee on Professional Recognition, reported that the qualifications for proposed uniform grades of membership in engineering societies had been formulated. These were accepted by ECPD at the meeting and are being submitted to the various societies with the hope of acceptance. Three grades of membership are recommended: Member, Associate Member, and Student Member; with the two optional grades of Fellow and Affiliate, if desired.

The Committee on Student Selection and Guidance and the Committee on Professional Training reported studies under way. The Committee on Principles of Engineering Ethics, represented by Wm. F. Ryan, vice-chairman, reported that the "Canons of Ethics" had been adopted during the year by the Engineering Institute of Canada, and that 56 societies, with a combined unduplicated membership of well over 100,000 engineers, had now

adopted the Canons, with endorsement by fifteen others. Of the leading societies, he reported that only AIME had taken no action, but this was remedied at the AIME Board meeting on Nov. 16, when AIME also approved the Canons. It is planned to publish them in the forthcoming Directory.

During the year ending Sept. 30, 1949, ECPD had an income of \$37,722 of which \$20,650 came from school inspection fees, \$12,287 in the form of grants from sponsoring organizations, and \$4,676 from publication sales. Expenses were \$36,535, of which the activities of the Committee on Engineering Schools (the accrediting agency) spent \$18,608, and the Committee on Information (for reports and reprints) \$1,796. Cost of other publications was \$8,051, and salaries, rent, and office expense totaled \$7,846. An office, under the direction of Miss Elsie Murray, is maintained on the 14th floor of the Engineering Societies Building in New York.

Officers for the coming year, elected at the annual meeting, are: Chairman, H. S. Rogers, president of the Polytechnic Institute of Brooklyn; Vice-Chairman, L. F. Grant, associate professor, Queens University, Kingston, Ont.; Secretary, C. E. Davies, secretary, ASME; and Assistant Secretary, W. N. Carey, secretary, ASCE. The Executive Committee consists of the foregoing, plus V. T. Boughton, C. E. Lawall, J. W. Parker, E. W. Davis, L. F. Grant, C. G. Kirkbride, H. T. Heald, and C. S. Crouse.

## AIME Annual Business Meeting

Notice is hereby given that the Annual Business Meeting of the AIME will be held at the Hotel Statler, New York City, at 4:00 p.m., Monday, February 13, 1950, instead of on the third Tuesday in February, as provided in the bylaws.

An executive session of the incoming Board of Directors will be held immediately following the Annual Business Meeting.

## AIME Dues Payable January 1

Pursuant to Article II, Section 2, of the Bylaws of the AIME, notice is hereby given that dues for the year 1950 are due on January 1 as follows:

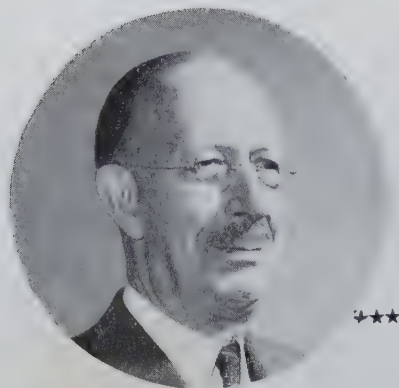
Members .....	\$20.00
Associate Members .....	20.00
Junior Members for the first six years of Junior Membership .....	12.00
Junior Members, thereafter .....	17.00
Student Associates, including an annual subscription to a journal .....	4.50
Student Associates, without journal subscription .....	2.00

Bills for dues were sent during October and November to members of the Metals and Petroleum Branches, and will be sent in December to members of the Mining Branch. Payment upon receipt of bill will be appreciated and will assure a prompt receipt of the journals upon publication in 1950.



# AIME

## Annual Meeting



**A** new high in the number and variety of technical papers, a greater number of technical sessions, and bigger and better things in the field of entertainment—all are forecast for the big AIME Annual Meeting, to be held February 12-16 in New York's Hotel Statler. Each of the ten Divisions of the Institute is planning one or more sessions, and six or eight concurrent sessions are likely to be the rule.

Local Section activities will receive particular attention when the Institute gathers, and Delegates will have ample opportunity to discuss Section functions with the Board of Directors. A Council of Section Delegates will gather on Saturday morning, Feb. 11, for an all-day session. The following day, Sunday, Section Delegates will meet with the Board of Directors in the morning, with discussions continuing through the afternoon if necessary. The educators will hold their customary sessions on Sunday afternoon and evening. All of these pre-Meeting conclaves are designed to avoid conflict with the technical sessions.

General Dwight D. Eisenhower, president of Co-

lumbia University, will be the featured speaker at a "Welcoming Luncheon" which will officially open the Meeting on Monday. The man who led our fighting forces to ultimate victory, and who now heads one of the world's largest educational institutions, will doubtless have an important message for the AIME Members who are helping to engineer the peace.

The Annual Business Meeting of the Institute will also be held the first day of the week-long gathering. Every AIME Member is cordially invited to that meeting. The incoming Board of Directors will meet in executive session that evening, while the rest of those attending will start to partake of New York's fabulous entertainment facilities. Cocktail parties, a dinner-smoker, and radio and theatre parties will be arranged in advance by diligent members of the Entertainment Committee. Visits to the Metropolitan Museum of Art and to United Nations headquarters have been arranged for the ladies' afternoon pleasure.

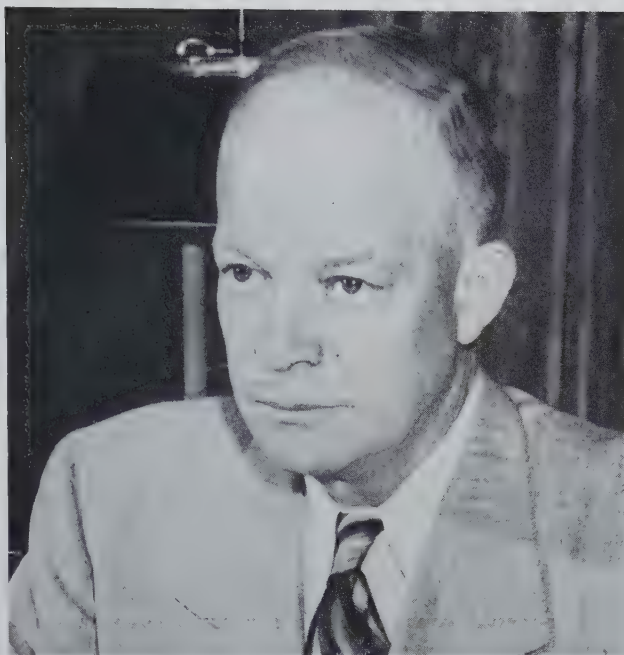
Many noteworthy sessions are being planned to attract Members' attention at the Hotel Statler.

*New York's gleaming, skyscraping Rockefeller Center, city within a city housing everything from golf schools to consulates. Meeting delegates will find everything here to interest and amuse them when they're not busy attending technical sessions in February.*





*Record-breaking array of sessions, papers, forecast for the big February meeting in New York. Bring your problems to numerous symposiums and seminars. Eisenhower to deliver keynote address. Special attention to Local Section activities planned. Bermuda cruise an added feature for post-meeting relaxation.*



*General Dwight D. Eisenhower, president of Columbia University, who will address the Welcoming Luncheon.*

The Minerals Beneficiation Division will sponsor a symposium on mill management during the week, and it is hoped that mill operators will bring their knotty problems and hard-won solutions to be aired for the benefit of all concerned. The Institute of Metals Division will have a particularly active program, beginning its meetings on Sunday, Feb. 12, and continuing with sessions during Monday, Tuesday, Wednesday and Thursday. The Extractive Metallurgy Division has scheduled seven sessions for the week, among which will be a symposium on fume and dust collection. The usual Student Forum will be held by the Mineral Industry Education Division, and is tentatively scheduled for Monday morning. One of the high lights of the Petroleum Branch program will be a seminar on economics, production technology, and personnel problems.

**Social Events.** On Tuesday, following the opening of technical sessions, social life will also begin in earnest, leading off with a luncheon and fashion show for the women in attendance. That evening is earmarked for an informal dance, but some members may wish to take advantage of the surprising number of tickets which have been secured for the hit show "South Pacific." The gala social event of the week will, of course, be the Annual Banquet, scheduled for Wednesday night. Mining companies will be more than usually liberal in supplying favors at the social functions this year. The M. A. Hanna Co. will provide the souvenirs at the Welcoming Luncheon, the American Zinc, Lead and Smelting Co. at the Smoker, and the American Smelting and Refining Co. at the Annual Banquet.

Petroleum men, the married ones that is, will not go unescorted to their Annual Dinner this year. In an unusual move, the committee in charge has elected to invite the ladies to this function.

**Cruise to Bermuda.** A nautical return to prewar meeting pleasures will be offered this year in the

form of a post-meeting cruise to Bermuda. The six day trip will include 2½ days on the island and 3½ days on the luxurious "Queen of Bermuda." The all-inclusive cost will be about \$180, or more if more elaborate shipboard accommodations are desired. Members and their families will thus have an opportunity to take and enjoy with congenial friends in the profession what is perhaps the most attractive short winter cruise out of New York. Leon V. Arnold, 36 Washington Square West, New York 11, N. Y., will gladly provide further information to those interested. **Write before Dec. 31.**

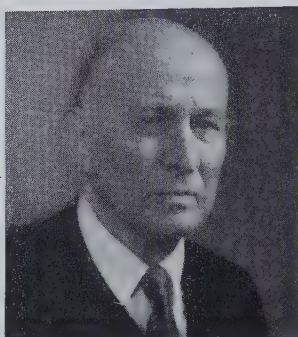
A meeting announcement, with a blank for reservations for the social events, will be mailed early in January. Members can assure themselves of tickets for the events of their choice with a minimum of inconvenience by returning the reservation cards with checks. Tickets will then be mailed up to Feb. 1, thereafter they will be held at the registration desk.

**More details** will appear in the January issues of the journals. Suffice it to say here that this will be the opportunity of the year to meet old friends in the profession and make new ones, to hear of the latest in technical progress in the mineral industry field, and what is perhaps most important, to meet informally, in just one trip, dozens of the people you would otherwise have to travel all over the country to see. In private conversation you can get the low-down on many points that never appear in print. Many boast that they have come to many Annual Meetings but have never attended a Technical Session—too much to learn outside the door! And outside of the hotel, New York has several legitimate theatres, a few movie palaces, a sports arena, an occasional night club, and on Fifth Avenue and Broadway some good stores. So give yourself a winter vacation and bring along your sweetheart or wife because this will be your last chance until 1952.



# AIME Medalists for 1950

Walter Hull Aldridge, president of the Texas Gulf Sulphur Co., was the guest of honor at a gala dinner at the University Club, New York City, on Nov. 16 on which occasion he was given the John Fritz Medal for 1950. The medal is customarily rotated annually between the AIME, ASME, ASCE, and AIEE, and is given for notable scientific or industrial achievement. Mr. Aldridge was cited "as engineer of mines and statesman of industry who, by his rare technical and administrative skills, has importantly augmented the mineral production of our country and Canada, and who, by giving unselfishly of his wisdom and vision, has guided his professional colleagues to higher achievements."



Walter H. Aldridge

L. E. Young, President of the AIME, presided at the dinner, and introduced Louis S. Cates, chairman of the John Fritz Medal Board, who told of the history and purpose of the medal. John R. Suman, substituting for Cornelius F. Kelley, who had suddenly been called to Montana because of the death of James R. Hobbins, told in his own inimitable way of the career and achievements of the medalist. Harvey S. Mudd presented the medal and certificate, which was gracefully accepted by Mr. Aldridge. In his acceptance speech he was most optimistic of the possibility of finding new ore bodies to supply the mineral wants of the United States, and of devising improved techniques for discovery and treatment. He said in part: "I believe that exploitable deposits in the world are far larger than many students of the subject seem to think. Mining engineers, with the indispensable help of physicists and engineers in other branches of the profession, will find these deposits and deliver the products to industrial users in such quantities as may be required. To doubt this is to underestimate the ability of the members of the great engineering profession."

Dr. Young closed the evening by reading numerous telegrams and letters that had been received, and called upon several of Dr. Aldridge's friends for informal remarks. Those responding included Norman B. Holter, John M. Lovejoy, E. De Golyer, Henry Krumb, Herbert E. Treichler, Herbert G. Moulton, Charles A. Wight, J. T. Kilbreth, H. O. C. Ingraham, and H. L. Smith. Erle Daveler handled the arrangements for the party.

A total of 77 had sent in their acceptances and every one was there. Besides those already mentioned, AIME members present included: E. G. Bailey, W. E. Wrather, D. H. McLaughlin, H. E. Dodge, A. J. McNab, H. K. Masters, A. L. Walker, Jr., F. E. Wormser, A. G. Wolf, R. F. Bacon, Clyde Williams, F. A. Ayer, S. H. Dolbear, E. C. Meagher, J. K. McCabe, M. L. McCormack, W. T. Lundy, J. H. R. Arms, H. DeWitt Smith, D. B. Gillies, L. E. Elkins, A. B. Kinzel, P. D. Merica, Bradley Stoughton, Wilber Judson, O. B. J. Fraser, J. F. Thompson, E. R. Weidlein, C. V. Millikan, H. R. Wemple, M. B. Gentry, Philip Kraft, J. C. Kinnear, R. H. Sales, T. B. Counselman, S. J. Swainson, and E. H. Robie. Also in attendance were friends of the Guest of Honor from among many in professions other than engineering.

Charles F. Rand Memorial Medal to F. H. Brownell, "For sound leadership in the administration of nonferrous mining and metallurgical enterprises and for outstanding contributions to society in financial and legal matters relating to the nonferrous metal industry."

James Douglas Metallurgical Medal to Francis C. Frary, "For distinguished achievement in science and contribution to society by broadening the field of knowledge in all phases of the aluminum industry, and for his notable success in directing a vast



F. H. Brownell

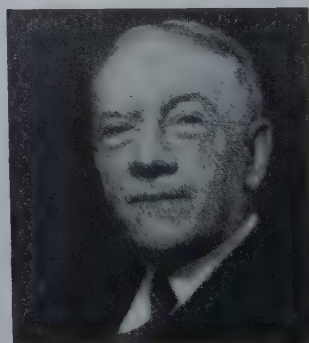
research project in this industry."

William L. Saunders Mining Medal to Howard N. Eavenson, "For the leadership he has contributed to the mining profession, and particularly for his engineering skill in design, construction and installation of productive facilities for the coal mining industry, coupled with his inspirational aid to other research and his thorough

and expressed knowledge of the economic problems of the coal industry."

Anthony F. Lucas Petroleum Medal to William E. Wrather, for his untiring leadership in guiding and developing the science of petroleum geology, for reducing this science to effective application in practice, and for the inspiration he has given to others in the profession by his dedication to public service."

Other medalists and award winners, with their pictures and citations, will be included in a later issue of the journals, probably in the January 1950 number; Erskine Ramsay Coal Medal to Paul Weir; R. H. Richards Award to A. F. Taggart; Rossiter W. Raymond Memorial Award to Walter R. Hibbard; Robert W. Hunt Silver Medal to John S. Marsh; Metals Division Annual Award to M. Cohen and W. J. Harris, Jr.



H. N. Eavenson



William E. Wrather



# Information and Advice to Members of the AIME Nominating Committee

Resolution Adopted by the Board of Directors on April 17, 1936 and Amended on May 21, 1947 and Nov. 16, 1949.

Recognizing the fact that the problems of the committee named by the Board to prepare the "official ticket" for officers and Directors of the Institute are various and difficult; and desiring to assist this committee by setting forth some of the principles that should guide the committee and some of the qualifications that should be required of candidates, the following suggestions are formulated and the Secretary is directed to publish them in the monthly journals concurrently with the publication of the names of each newly appointed Nominating Committee and to send a copy to each member of the Committee.

**Nominations to Be Made.** Each year nine Directors are nominated. One of these nine Directors serves as President for one year, then two years as Past President, and two serve as Vice-Presidents for their entire term of three years. It is the duty of the Nominating Committee to nominate eight Directors including those who are to serve as President and Vice-Presidents. The ninth Director is nominated by the Board of Directors.

In the event that a nominee for President or Vice-President is already a Director, whose term will not have expired when he takes his new office, it will be the duty of the Board in accordance with Article VI, Section 3 of the Bylaws to elect a successor to fill his unexpired term as Director. The Nominating Committee need therefore give no consideration to this question in case their nominee for one of these offices is already a Director. A Director may be nominated to succeed himself.

**Geographical Distribution.** The Bylaws provide that "the Nominating Committee shall, so far as practicable, distribute the representation on the Board geographically." To assist the Nominating Committee in carrying out this provision of the Bylaws and to encourage, so far as will not conflict with other necessary considerations, a consistent basis for such distribution from year to year, the United States, Canada, and Mexico have been divided into fifteen districts, as shown on the official map. (See page 66, 1948 Directory.) On the basis of number of members, Districts 1, 5, 6, 11, 12, 13, 14, and 15 would be entitled to one Directorship each; District 2, three Directorships; District 3, eight Directorships; 4 and 10, two Directorships each; and Districts 7, 8, and 9, two Directorships between them. This leaves two Directorships at large.

Attention is called to the fact that many Institute members now resident in New York formerly lived in the West, South, or North and still have intimate business connections that compel them to visit various parts of the country periodically. Suitable choice of such candidates may assist the Committee in attaining a well-balanced geographical distribution. Such men are particularly valuable as Directors and their nomination and election tends to minimize the objection that a large number of Directors reside in New York.

**Consultation with Local Sections.** In order that the members residing in a District may be given an opportunity to express preference as to their representative or representatives, the Nominating Committee should invite the Local Sections in each District to suggest candidates for the consideration of the Committee. While the Committee may not always find it practical to nominate a person so proposed, such endorsement should be given primary consideration by the Nominating Committee.

**Distribution as to Major Interest.** In addition to geographical distribution of Directorships it is highly desirable that the Board should include representatives of all the various phases of Institute activity. Each of the technical Divisions is entitled to representation by one or two Directors whose major interest is in its field, and so far as practicable and consistent with other considerations, such representation should be proportional numerically to the membership in these Divisions. With the same end in view, it is desirable that the Directors should represent diverse fields, for example, geology, iron mining, nonferrous metallurgy, economics, and ore dressing. In seeking to maintain appropriate technical Division and group representation on the Board, the Nominating Committee may seek and solicit the suggestions of individual Divisions or groups.

The Committee may note in this connection that the Chairman of each Division is a voting member ex officio of the Board of Directors during his term in office.

In nominating the Candidate for Director and President, particular consideration should be given to "major interest." It is desirable that the principle of rotation as to primary interest should be a factor in selecting the Presidential candidate.

**Obligations of Directors.** In establishing definite geographical Districts for the selection of Directors, it is the purpose and hope of the Board that an effective liaison between the Board and all sections of the country can be maintained. To this end, each Director will be considered as representing a geographical District. This arrangement, in conjunction with representation of the Divisions, will be effective only to the extent that the Director actually functions in behalf of his District or his Division. It is the duty, therefore, of the Nominating Committee to make it clear to the prospective nominee that, in addition to the honor, the position of Director carries with it definite responsibilities. The Committee should receive from the Local Sections suggesting candidates, or otherwise obtain assurance from the candidate that he is able and willing to undertake these responsibilities.

A Director should plan to attend at least two meetings of the Board, and as many other meetings as is practical, taking into account the distance he resides from New York; he should keep himself informed of the activities of the technical Division and Branch that represents his major interest and should attend meetings of that Division when practical; and he should keep in close touch with the officers of the Local Section or Sections in his district by attending meetings of the Sections or otherwise and should assume a personal responsibility for promoting healthy Local Section activities.

The Board offers this Information and Advice to assist the Nominating Committee in its functions but points out that Article IX, Section 1, paragraph 4 of the Bylaws which reads: "This Committee shall proceed to the selection of candidates and the naming of a ticket. In making such selections the Nominating Committee, shall, so far as practicable, distribute the representation on the Board geographically, provided that seven Directors shall be residents of New York City or the territory within a radius of fifty miles of the headquarters of the Institute" is the mandatory instruction to the Committee and that the interpretation of this paragraph and matters outside its intent are left to the discretion of the Committee.



## NOMINATING COMMITTEE FOR OFFICERS FOR 1951

### Members at Large

O.B.J. Fraser, Chairman—Assistant Manager, Development and Research Division, International Nickel Co., 67 Wall St., New York, N. Y.

Paul E. Fitzgerald—Dowell Incorporated, 524 Kennedy Bldg., Tulsa 3, Okla.

P. D. I. Honeyman—Inspiration Consolidated Copper Co., Inspiration, Ariz.

### Local Section Representatives

BOSTON—Raymond B. Ladoo, 42 Huntington Road, Newton, Mass.

(Canada)—H. M. Griffith, Works Mgr., Steel Co. of Canada, Ltd., Hamilton, Ont.

CENTRAL APPALACHIAN—Charles T. Holland, Virginia Polytechnic Institute, Blacksburg, Va.

DELTA—F. E. Simmons, Jr., Houma, La.

DETROIT—Roger F. Mather, Chief Metallurgist, Kaiser-Frazer Corp., Willow Run, Mich.

LEHIGH VALLEY—E. A. Anderson, New Jersey Zinc Co., Palmerton, Pa.

NEW YORK—T. B. Counselman, The Dorr Co., 570 Lexington Ave., New York 22, N. Y.

NEVADA—Louis D. Gordon, Supervisory Engineer, State of Nevada, Mining Section, RFC, Reno, Nev.

OREGON—Pierre R. Hines, 1007 E. Burnside St., Portland 14, Ore.

SOUTHERN CALIFORNIA—Walter B. Hester, 294 St. Albans Ave., S. Pasadena, Calif.

SOUTHEAST—W. C. Chase, General Superintendent of Mines, Alabama Byproducts Corp., Birmingham, Ala.

SOUTHWESTERN NEW MEXICO—David W. Boise, 917 West St., Silver City, N. Mex.

SOUTHWEST TEXAS—R. C. Granberry, Jr., Humble Oil & Refining Co., Corpus Christi, Texas.

TRI-STATE—George M. Fowler, 608 Joplin National Bank Bldg., Joplin, Mo.

UPPER PENINSULA—Grover J. Holt, Cleveland-Cliffs Iron Co., Ishpeming, Mich.

UTAH—B. E. Grant, U. S. Smelting Refining and Mining Co., 1119 Newhouse Bldg., Salt Lake City, Utah.

## Coming Meetings

### DECEMBER

- 2 Columbia Section, AIME.
- 4-7 AIChE, national meeting, Pittsburgh, Pa.
- 5 Boston Section, AIME.
- 6 Society for Applied Spectroscopy, New York City.
- 7 American Mining Congress, Annual Business Meeting, New York City.
- 7 Chicago Section, AIME. W. C. Schroeder on synthetic liquid fuels.
- 7 New York Section, AIME. L. E. Young on research in coal mining.
- 8-10 Seventh Annual Conference, Electric Furnace Steel Committee, Iron and Steel Division, AIME, Hotel William Penn, Pittsburgh.
- 9 Rio de Janeiro Section, AIME.
- 9 St. Louis Section, AIME, York Hotel.
- 13 Delta Section, AIME. W. H. Skinner on gas condensate well corrosion problems.
- 13 East Texas Section, AIME.
- 14 Connecticut Section, AIME. F. H. Wilson on intergranular parting of 70:30 brass.
- 14 El Paso Metals Section, AIME.
- 14 San Francisco Section, AIME.
- 15 Carlsbad Potash Section, AIME.
- 15 Utah Section, AIME.
- 16 Oregon Section, AIME.

- 19 Detroit Section, AIME.
- 20 Gulf Coast Section, AIME.
- 20 Washington, D. C., Section, AIME.
- 21 Southwest Texas Section, AIME.
- 26 Alaska Section, AIME.
- 26-31 AAAS, Penn zone hotels and Columbia Univ., New York City.
- 27 Montana Section, AIME.

### JANUARY 1950

- 2 Boston Section, AIME.
- 4 Chicago Section, AIME. T. B. Counselman on Fluidization of Solids in Noncatalytic Operations.
- 6 Columbia Section, AIME.
- 10 Delta Section, AIME. Water injection.
- 10 East Texas Section, AIME.
- 11 El Paso Metals Section, AIME.
- 11 San Francisco Section, AIME.
- 11 Southwestern New Mexico Section, AIME.
- 13 Rio de Janeiro Section, AIME.
- 16 Minnesota Section, AIME. Annual meeting, Hotel Duluth, Duluth, Minn., Mining symposium conducted by the Center of Continuation Study, of the Univ. of Minn. follows annual meeting on Jan. 17 & 18.
- 16-19 Plant Maintenance Show, 4-day exposition and conference, Auditorium, Cleveland, Ohio.
- 18-20 American Society of Civil Engineers, annual meeting, New York.
- 30-Feb. 3 AIEE, winter meeting, Hotel Statler, New York.

### FEBRUARY 1950

- 10 Southwestern Section, Open Hearth Steel Committee, Iron and Steel Division, St. Louis, Mo.
- 12-16 Annual Meeting, AIME, Statler Hotel, New York City.

### APRIL 1950

- 4-7 Nat'l Assn. of Corrosion Engineers, St. Louis.
- 10-12 Open Hearth Conference, and Blast Furnace, Coke Oven and Raw Materials Conference, Netherland Plaza Hotel, Cincinnati.
- 19-21 American Society of Civil Engineers, spring meeting, Los Angeles.
- 23-26 American Ceramic Society, annual meeting, New York City.
- 24-26 AMC Coal Convention, Netherland Plaza Hotel, Cincinnati, Ohio.
- 25-26 Annual Metal Powder Show, Book-Cadillac Hotel, Detroit.

### OCTOBER

- 21-27 National Metal Congress, Chicago.

### DECEMBER 1950

- 7-9 Electric Furnace Steel Conference, Iron and Steel Div., Hotel William Penn, Pittsburgh.

### APRIL 1951

- 2-4 Open Hearth and Blast Furnace, Coke Ovens and Raw Materials Conference, Iron and Steel Division, Statler Hotel, Cleveland.



## Conditions Under Which 1950 Publications Are Available to Members

Pursuant to Article X of the By-laws of the AIME, the following information is hereby given as to the "conditions, prices, and terms under which the various classes of members, and Student Associates, severally, shall be privileged to obtain publications of the Institute during the ensuing year."

Publications authorized for 1950 publication include the following:

**Mining Engineering**, published monthly, containing material, including technical papers, of interest to those engaged in exploration, mining geology and geophysics, and metal, nonmetallic, and coal mining and beneficiation, and fuel technology.

The **Journal of Metals**, published monthly, containing material, including technical papers, of interest to those engaged in nonferrous smelting and refining, iron and steel, and physical metallurgy.

The **Journal of Petroleum Technology**, published monthly in Dallas, containing material, including technical papers, of interest to those engaged in petroleum production.

Current copies of any one of the above journals will be supplied to all members in good standing without further charge. (A member ceases to be in good standing if current dues are not paid by April 1.) If more than one of the monthly journals is requested, \$4 extra will be charged for an annual subscription, or 75 cents for single copies. The nonmember subscription price for each journal is \$8 in the Americas; foreign, \$9. Student Associates who pay dues of \$4.50 will be entitled to the same privileges for all publications as members except as noted below; those who pay \$2 will not receive individual subscriptions to the journals but otherwise have the same privileges.

Three volumes of "Transactions" are authorized for 1950 publication, as follows: No. 184, Mining Branch; No. 185, Metals Branch; and No. 186, Petroleum Branch. These volumes will be available to paid-up members at \$3.50 each for a first copy. Nonmembers \$7 in the United States; foreign \$7.50.

Special volumes now planned for publication in 1950 include the following:

(1) Open Hearth Proceedings. Price to AIME members, \$7.50; to students in accredited colleges in the United States, \$3.

(2) Blast Furnace, Coke Oven, and Raw Materials Proceedings. AIME members \$5; students as above, \$2.50.

(3) Electric Furnace Steel Proceedings. AIME members \$7; students as above, \$3.

(4) Symposium on Tube Production Practice. Price to be set.

(5) Basic Open Hearth Steelmaking, 2d ed. Price to be set.

(6) Statistics of Oil and Gas Development and Production. AIME members \$3; nonmembers \$6.

(8) Conservation of Petroleum, by Stuart Buckley. Date of publication and price to be determined.

(9) Coal Preparation, 2d ed. Price to members \$4 for one copy; additional copies at nonmember price of \$8.

If dues are paid subsequent to Jan. 31, back issues of Institute publications will be supplied only if adequate stocks are on hand. A member may not receive a volume of "Transactions" or a special volume in lieu of a monthly journal, free of charge on membership. Members in arrears for dues are not entitled to special members' prices for publications.

Rocky Mountain Members may have their choice of an annual subscription to one of the monthly journals on request.

## What Went on at Recent Local Section Meetings

SECTION	DATE	ATTENDANCE	SPEAKER, AFFILIATION, AND SUBJECT
Alaska.....	Oct. 24	22	H. G. Wilcox, RFC, on history and purpose of the RFC.
Black Hills.....	Sept. 16	51	N. Herz, Homstake Mining Co., on postwar metallurgical problems.
Boston.....	Oct. 3	68	R. L. Nichols, Tufts College, on the Arctic and Antarctic.
Carlsbad Potash.....	Sept. 15	85	L. E. Young, President, AIME, on mining aspects of the ECA.
Carlsbad Potash.....	Oct. 20	50	A. G. Wolf, Texas Gulf Sulphur Co., on sulphur from sulphur domes.
Chicago.....	Sept. 14	85	Sir Charles Goodeve, British Iron and Steel Research Assn., on organization and work of the Association.
Colorado.....	Oct. 11	86	L. E. Young, President, AIME.
Columbia.....	Oct. 7	13	J. P. Spielman, Washington State College, on curriculum for mining schools.
Connecticut.....	Oct. 12	32	A. Phillips, Yale Univ., on metallurgy in Brazil.
El Paso Metals.....	Sept. 13	82	L. E. Young, President, AIME, on technical assistance to foreign countries. A. C. Serrano, Mexican official, on mining industry of Mexico.
Gulf Coast.....	Sept. 20	115	A. W. Bounds, Stanolind Oil & Gas Co., on legal and political history of the Tidelands controversy.
Kansas.....	Sept. 7	62	J. E. Smith, Johnson Oil Field Service Co., on drill stem testing in open hole.
Lehigh Valley.....	Sept. 7-8	43	Annual inspection trip, N. J. Zinc Co. mine and mill.
Lehigh Valley.....	Oct. 21	84	J. E. M. Wilson, Jeffrey Mfg Co., on mechanical mining of bituminous coal.
Montana.....	Oct. 13	110	L. E. Young, President, AIME.
North Pacific.....	Sept. 22	51	E. A. Cocanower and L. Heinzinger, Bethlehem Pacific Steel Corp., on the history of Western steel industry, and purchasing raw materials for a Seattle plant, respectively.
North Pacific.....	Oct. 17	60	L. E. Young, President, AIME.
New York.....	Sept. 7	112	E. Just, ECA, on current strategic materials development in the ERP.
New York.....	Oct. 13	103	M. L. Haider, Imperia Oil, Ltd., on Canadian oil developments.
Ohio Valley.....	Sept. 27	.....	G. von Stroh, BCR, on an analysis for a continuous mining machine.
Ohio Valley.....	Oct. 13	35	B. D. Blackie and B. W. Dunnington, students, Ohio State Univ., on looking Labrador over, and corrosion at elevated temperatures, respectively.
Pennsylvania-Anthracyte	Oct. 28	180	J. R. Hoffert, dept. of health, Commonwealth of Pa., on abating stream pollution.
Rio de Janiero.....	Sept. 8	.....	A. I. de Oliveira, National Petroleum Council, on petroleum and refineries in Bahia.
Rio de Janiero.....	Sept. 22	.....	L. J. de Moraes, Cia. Aeos Especiais de Itabira on company activities and objectives.
Rio de Janiero.....	Oct. 13	.....	S. F. Abreu, Brazilian delegate to UNSCOUR, on meeting and visit to oil shale plant.
Southwest Texas.....	Sept. 14	233	J. L. P. Campbell, Lane Wells Co., on the correlation of gamma ray and neutron radioactive well logs.
Southeast.....	Oct. 4-5	96	L. E. Young, President, AIME.
Southeast.....	Oct. 21	119	A. C. Fieldner, USBM, on coal and its utilization.



# Personals

**Robert E. Baarson** is working for the Eagle-Picher Co. as an engineer in exploratory research. His address is 2512 Main St., Joplin, Mo.

**T. H. Barrett**, formerly associate professor at Robert College, Istanbul, Turkey, is teaching at Michigan College of Mining and Technology, Houghton. He was recently reelected a director of the Construction Men's Association of New York City, but expects to have time for some consulting work.

**Oliver W. Borgeson**, a '49 graduate of Michigan College of Mining and Technology, is sales representative in the Lake Superior district for the Mine Safety Appliances Co. His address is 617 N. 10th Ave. E., Duluth 5.

**Everett O. Bracken** received his B.S. degree in geological engineering from the Montana School of Mines in June and is employed by the Silver King Coalition Mines Co. at Park City, Utah, as a junior geologist.

**Andre L. Brichant** has taken up his duties as manager of the Belgian Government trade exhibition in Kansas City, Mo. The exhibition opened at the end of October and will be of a permanent character.

**Joseph F. Brown** worked last summer for the Lehigh Navigation Coal Co. and then returned to Columbia to study for an EM degree under a Krumb Fellowship.

**Charles H. Burgess**, mining consultant and geologist, succeeded **Evan Just** as director of the strategic materials division of ECA on Nov. 14. After more than sixteen months as director, Mr. Just returned to the editorship of E&MJ. Mr. Burgess had been deputy director of the ECA division since October 1948. He had worked for Anaconda and the Quartz Hill Mining Co. and during the war was with the BEW, OPA, and WPB.

**Donald Carlisle** is a lecturer in the department of geology at the University of California at Los Angeles.

**D. J. Christie** has left S. W. Africa, where he was a metallurgical engineer for the Tsumeb Corp., to become a student at the Harvard Business School.

**A. F. Crosby** recently took the post of mill superintendent with the New York and Honduras Rosario Mining Co., San Juancito, Honduras.

**James W. Crosby, III**, recently took the post of junior mining geologist with the California State Division of Mines and Geology. His address is 543 Ramsell St., San Francisco 27.



Herbert Hoover

**Herbert Hoover** received the Frederick W. Taylor Key of the Society for Advancement of Management on Nov. 3, in recognition for his services to the nation as chairman of the Commission on Organization of the Executive Branch of the Government. The key is presented annually for the outstanding contribution to the advancement of the art and science of management as conceived by F. W. Taylor, pioneer of scientific management.

On Feb. 10 Mr. Hoover will be one of the recipients of the 10th annual Moles award. The Moles, composed of men now or formerly engaged in the construction of tunnel, subway, sewer, foundation, marine, subaqueous, or other heavy construction, honor one member and one nonmember for their contributions to the construction field and for their achievements as citizens. Mr. Hoover is the 1950 nonmember recipient.

**Erle V. Daveler**, vice-president and director of the American Zinc, Lead and Smelting Co., married Mrs. Lola Bullard at the Church of St. Ignatius Loyola, New York City, on Oct. 27. Mr. Daveler, a director of the AIME, is chairman of the Institute's Finance Committee.

**C. B. E. Douglas**, after spending most of the summer in Alaska, returned to the Boston office of the U. S. Smelting Refining and Mining Co., which is now his headquarters.

**Johannes S. Enslin** is inspector of

mines, Department of Mines in Johannesburg, S. Africa.

**A. W. Estey** can now be addressed in care of Nicholson Mines Ltd., Box 42, Fort McMurray, Alta.

**K. R. Fleischman** is mining engineer and metallurgist for Dickson Primer & Co., Pty., 73 Day St., Sydney, N.S.W., representing Metal Traders Ltd., London, and Metal Traders Inc., New York, in their metal and ore buying interests throughout Australasia.

**J. F. Frost** left the Northern Peru Mining and Smelting Co. in Trujillo to go to Buchans, Newfoundland, where he can be reached in care of the Buchans Mining Co.

**E. D. Gardner** took up permanent duties at Washington, D. C., on Oct. 20 as chief mining engineer with the Bureau of Mines. He had been in Denver, working on oil shale.



Walter R. Brown

**Walter R. Brown** has bought a home at 3320 NW 4th St., Miami 35, Fla., in which to house his family while he is away. At the moment he is not engaged in mining work but has hopes of some for the future.

**I. G. Irving**, mining engineer of Butte, and his associates are carrying on active development work at three properties in Montana which were prominent a number of years ago, the Pyrenees, the Spar, and the Norwich mines.

**Dean D. Kerr** graduated from the University of Kansas in August and is employed by the Utah copper division of the Kennecott Copper Corp.



His address is 55 2nd Ave., Midvale, Utah.

**Edward J. Lee** can be reached at 746 Madison Ave., Scranton, Pa. He is an engineer with the Hudson Coal Co.

**Hsin Te Lee** is working for the Grandview Mine at Metaline Falls, Wash., as assayer.

**Paul Linz** has been made chairman of the board of directors of the South American Minerals and Merchandise Corp. He continues as personal assistant to **Mauricio Hochschild** in all his enterprises. SAMINCORP represents Hochschild interests in Peru, Bolivia, Chile, Argentina, and Brazil.

**Orville R. Lyons** left Battelle Memorial Institute on Nov. 1 to become coal preparation engineer with Heyl & Patterson, Inc., Pittsburgh.

**Vernon L. Mattson**, former chief engineer for the Consolidated Feldspar Corp., is director of the Colorado School of Mines Research Foundation. His home address is 1198 Pikeview St., Lakewood Station, Denver.

**G. C. McCartney** recently moved his consulting office to 15 King St. W., Toronto 1, Ont. He is devoting considerable time to the search for uranium deposits, especially in the Lake Athabaska area of Northern Saskatchewan.

**L. Charles McHenry**, who graduated from Missouri School of Mines last June, is an engineer with the Tennessee Coal, Iron and Railroad Co. His address is 2208 Clarendon Ave., Bessemer, Ala.

**Edward C. Meagher**, treasurer of the Texas Gulf Sulphur Co., has been re-elected president of United Engineering Trustees. **James L. Head**, Chile Exploration Co., was re-elected assistant treasurer and **John H. R. Arms** continues as secretary.

**William L. Merritt** has opened a consulting mining engineering office at 1108 Hobart Bldg., 582 Market St., San Francisco 4, and has discontinued both the Ohio and Sanger, Calif., addresses.

**Melbourne W. Miller** graduated from the Colorado School of Mines last May and is employed by the Euclid Road Machinery Co. of Cleveland, Ohio, as a field engineer.

**R. C. Morgan** is working as a junior engineer for the Columbia Iron Mining Co. at the Iron Mountain mine. His address is 171 S. 100 E., Cedar City, Utah.

**Frank Noe**, of the Bureau of Mines, has been transferred from the Salt Lake City office to Rio de Janeiro where he has been assigned to work with the Departamento Nacional da Producao Mineral of the Brazilian government.

Just thirty years ago, when Horace Winchell was President of the AIME and Herbert Hoover had been selected as the President for the ensuing year, **Grace Pugsley**, then barely out of kindergarten, applied for a job at Institute headquarters and got it. In addition to the stenographic competence which is possessed by most young ladies entering business life, she brought to her new job, as it proved, a natural interest and ability in both the editorial and advertising phases of the publishing business; a cheerfulness that enabled her to get along with even the most difficult advertising managers (editors are usually not troublesome); and an in-



born desire and energy to do the job before her as well as possible, with no complaint about occasional overtime or the extra assignments that fall to the lot of a willing worker.

In the years since 1919 most of Grace Pugsley's work has been in one capacity or another on the magazines that the Institute publishes, and in recent years she has been secretary and assistant to the advertising manager of Mining and Metallurgy and Mining Engineering and production manager of these journals and of the annual Directory. Having served longer than any one else on the AIME staff, she has often been its spokesman and representative, and naturally is one of the chief figures in such social events as the Christmas staff party. Five years ago she became Mrs. E. F. Stickle. Two other young ladies who joined the Institute staff within a year after Grace Pugsley—Grace Townsend and Irene Klein—are also still on the roll.

The Secretary, who joined the Institute as a Member the same time that Grace did as an employe unites with the staff in congratulating her on

thirty years of faithful and highly competent service. She is the kind of person with whom we have all enjoyed working.



Sidney J. McCarroll

**Sidney J. McCarroll** resigned as manager of Minerales de Nacozari on Nov. 1. This resignation coincides with the withdrawal of American interests from the company, which is now Mexican controlled. His present address is 1446 12th St., Douglas, Ariz.

**Thomas B. Rees** has taken over the Charleston-Logan, W. Va., territory for the Goodman Mfg. Co. as sales engineer. He had been in the Pittsburgh and Huntington territories. On Oct. 3 he moved his family from Signal Mt., Tenn., to 9 Estill Drive, Charleston, high above the Kanawha River. Mr. Rees has also been associated with the Ross Meehan Foundries, Hedges Mfg. Co., Johns Manville, and Combustion Engineering.

**Stuart St. Clair** returned to New York in October after "20,000 miles over Africa" by air safari. He represented Overseas Consultants, Inc., of New York City, who, in conjunction with the engineering firm of Sir Alexander Gibb & Partners of London, conducted a reconnaissance survey of a proposed connecting link between the Rhodesian and East African Railway Systems for the British Colonial Office. The trip took three months and included all of Tanganyika and much of Kenya, Nyasaland, and N. and S. Rhodesia, and a little of Belgian Congo and Portuguese East Africa.

**Robert T. Sanden**, recent graduate of the University of Minnesota, is an assistant research engineer for the M. A. Hanna Coal and Ore Co. He can be reached at 2623½ 1st Ave., Hibbing, Minn.

**Donald J. Saunders** is attending the University of Minnesota graduate school, working for an M.S. degree in geology.



**Carl W. Sawyer** is a metallurgist at King Lease Inc., Ouray, Colo. The mine, located about six miles from Ouray, produces lead, zinc, gold, and silver.

**Alan A. Sharp**, stationed with the Bureau of Mines health and safety division at Phoenix, was recently transferred to Denver, which has just been designated as regional headquarters for the Bureau's Rocky Mountain area. Having cut his eye teeth in mining around Colorado, he finds the change welcome and anticipates good skiing within short driving distance from the mile-high city.

**Philip J. Shenon** is now in Salt Lake City filling the post of chairman of the division of mineral engineering at the School of Mines of the University of Utah.

**Robert H. Shepard** has accepted the post of company geologist with Malco Refineries, Inc., of Roswell, N. M. He had been with the U. S. Geological Survey.

**Daniel C. Shewmon** has become an instructor with the mining department of West Virginia Institute of Technology, Montgomery, W. Va., after working nearly five years with the New Jersey Zinc Co., both in New Jersey and Virginia.

**John P. Skinner** has been transferred from Franklin, N. J., by the New Jersey Zinc Co. to the Eagle mine in Gilman, Colo., as assistant mine foreman.

**Alex J. Speal** has been employed as assistant mine engineer by the Richard Ore (Iron) Co., Wharton, N. J., since graduating from Lafayette College as a mining geologist in June. His address is Box 243, Mt. Hope Rd., Rockaway Township, N. J.

**Hugh S. Spence** has retired from the Mineral Resources Division, Canadian Bureau of Mines, after 39 years of service. The Bureau's specialist in a wide range of industrial minerals, he is also an authority on radioactive minerals having made the first official report on the pitchblende discoveries at Great Bear Lake in 1931. His *Prospector's Guide to Uranium and Thorium Minerals* has had the widest circulation of any publication put out by the Bureau. Now he intends to engage in private consulting practice, with an address at 222 Argyle Ave., Ottawa, Ont.

**Harvey W. Smith** graduated from the Colorado School of Mines last May and is now employed by the Magma Copper Co., Superior, Ariz. Mail reaches him at P. O. Box 587, Superior.

**Ernest M. Spokes**, after acquiring a M.S. degree in mining engineering in

August, was made assistant professor of mining engineering at the University of Kentucky on Sept. 1.

**Edward Steidle, Jr.**, is in Paris as an engineer adviser on mine mechanization for the Joy Mfg. Co.

**W. R. Storms** has been transferred by the Bureau of Mines from Silver City, N. Mex., to Tucson, Ariz., where his address is 4020 E. Whitman St.



Rinaldo V. Taborelli

**Rinaldo V. Taborelli** went to the New Mexico School of Mines in October as development contract officer at the school, whose research and development division has a contract with the Bureau of Ordnance. Along with his regular duty, the present assignment affords interesting contact with his neglected profession—mining.

**G. Douglas Strachan** has been with the United States, Philippine War Damage Commission in Manila since July, 1948. At the present time he is in charge of the mining section, with the full responsibility of appraising and adjudicating all mine damage claims.

**Weston Thomas**, formerly with the Potash Co. of America, is now with the Climax Molybdenum Co., New York City. His new residence address is 191 Main St., Southport, Conn.

**George A. Thompson, Jr.**, has been made assistant professor of the Stanford School of Mineral Sciences.

**Verle B. Utzinger**, former student at the University of Illinois, is now field engineer for the Muller Co., Decatur, Ill.

**John Vanderwilt** spent last summer in Norway as a consultant for the Strategic Materials Division of the ERC. He traveled extensively in Norway, examining the iron deposits of Sydvaranger near Kirkenes and the

Dunderland deposits, north east of Mo i Rana. The concentrating plant at Kirkenes was completely destroyed in 1944 by the retreating Germans and much of the destruction is still evident although a large part of the town has been rebuilt. Reconstruction of the plant is underway, aided by ECA funds, with partial completion hoped for in 1951. A steel plant is under construction at Mo i Rana. Pyrite ores are probably the most important; Mr. Vanderwilt saw representative deposits. Of the mica, nickel, copper, cobalt, manganese, graphite, and a few nonmetallic minerals, graphite is the most important at the moment. Norway has not had, and still is not having, an easy time, but they are working hard and no doubt will rebuild the forty percent of their industrial wealth destroyed by the war.

**Thomas R. Van Fleet**, who graduated from the University of Arizona last May, is working for the U. S. Vanadium Corp., in Uravan, Colo., as a mining engineer.

**Howard L. Waldron** is working on a training program with the Buas Drilling Co. His new address is 2331 Parkway, Bakersfield, Calif.

**Stanton Walker**, director of engineering of the National Sand and Gravel Association, Washington, D. C., announces the removal of the Association's laboratory to its new location, Building H of the Martin Engineering College, Washington. Extensive new equipment and facilities are being provided.

**Robert R. Wallace** has completed his studies at the Michigan College of Mining and Technology and is working as a mining engineer with the Oliver Iron Mining Co. in Hibbing, Minn.

**Paul Weir**, president of the Paul Weir Co., mining engineers and geologists, Chicago, recently spent several weeks inspecting coal mining developments for the Turkish government. He supervised five American mining engineers, geologists, and management consultants who reviewed plans and specifications for modernization of key mines in the Turkish Zonguldak field with American equipment and methods. Before returning to this country, Mr. Weir toured mines in Great Britain to check progress of test installations of American equipment and methods in British mines.

**Reed F. Welch** succeeded **Brent N. Rickard** as manager of the Tucson ore-buying office of the American Smelting and Refining Co. on Oct. 1. Mr. Rickard, because of recent illness, is retiring from active duty but will remain in an advisory capacity until July 1, 1950. Mr. Welch has had ex-



tensive experience in the purchase of ores and concentrates, having acted as ore buyer in the Company's Salt Lake City office since 1937.



Howard J. Vander Veer

**Howard J. Vander Veer** has been appointed chief of the division of adjudication in the Colorado-Utah region of the Bureau of Land Management. He is responsible for all BLM mineral activities in the area, and supervises all homestead, rights-of-way, recreation, and other uses of the public lands administered by the BLM. He is the first mining engineer who has ever been appointed head of a regional division in BLM.

**Sam West** is doing graduate work in geology and mineralogy at the University of Arizona, Tucson.

**Jerry F. Whalen**, upon graduation from the Colorado School of Mines last July, took the job of mining engineer in the coal division of Eastern Gas and Fuel Associates and is located at Helen, W. Va.

**Glen B. Wilson** has the job of mine superintendent with the Crown Point Mining Co. His address is Box 761, Globe, Ariz.

**Harry J. Wolf**, mining and consulting engineer, has established his office at 420 Madison Ave., New York City.

**Rudolph G. Wuerker** has the post of assistant professor in mining at the University of Illinois, Urbana.

**Charles Will Wright** sailed for Europe in October. He will visit Athens, Rome, Paris, Madrid, Lisbon, and London, and can be addressed in care of the American Embassies in these cities. Besides getting information on certain mine development projects, he will make a study of the present mining legislation, exchange controls, labor conditions, taxes and tariffs, and the changes that are being considered to encourage better co-opera-

tion between local and foreign mining interests for increased output of strategic minerals under private enterprise.

## —In the Metals Branch—

**Robert W. Bohl** has been made assistant professor in the department of mining and metallurgical engineering at the University of Illinois.

**Gerard H. Boss** left the aeronautical materials lab., metallurgical division, of the Philadelphia Naval Base last May to join the metallurgy division of the Oak Ridge National Lab., Oak Ridge, Tenn.

**W. L. Brown** lives at 928 7th Ave. N.W., Great Falls, Mont. He is president and general manager of the General Distributing Co. in the oxy-acetylene and electric welding supply business with distribution throughout central Montana.



H. J. Kozlowski

**H. J. Kozlowski**, for the past 4½ years a research metallurgist in the physical metallurgy division of the Bureau of Mines at Ottawa, is on leave of absence to take graduate studies in physical metallurgy at Yale.

**C. L. Clayton** became affiliated with Geo. D. Clayton and Sons, Insurance, of Hannibal, Mo., on Nov. 1.

**Warren P. Chernock**, formerly a student at Columbia School of Mines, is now in the X-ray section of the metallurgy division of the Argonne National Lab. He lives at 311 S. West Ave., Elmhurst, Ill.

**A. A. Conrad, Jr.**, has been transferred by the Lorain Works of the National Tube Co. to the U. S. Steel Corp. of Delaware research laboratory for a period of training in research work. He is addressed at the lab., Lincoln Highway, Kearny, N. J.

**George S. de Moraes** graduated from Carnegie Tech last June and is now working as a metallurgical engineer at the Air Depot of the Brazilian Air Force in Sao Paulo.

**Paul Dixon, Jr.**, after completing work for his M.S. degree in metallurgy at Columbia, joined the metallurgical research department of the Chrysler Corp. in Detroit.

**W. E. Duckworth** has become research investigator with the Glacier Metal Co., Alkerton, Wembley, England.

**Sanford A. Estes** is chief metallurgist for the Hoyne Iron and Steel Co. He can be reached at 4901 Drexel Blvd., Chicago.

**Henry E. Frankel** recently took the job of research assistant at the metals research lab. of the Carnegie Institute of Technology. His present address is 4016 Saline St., Pittsburgh 17.

**Arthur E. Franks** is project manager of the metals research department of the National Research Corp., 70 Memorial Drive, Cambridge 42, Mass.

**Thomas E. Groce** is employed by the Kaiser Aluminum Co. at Newark, Ohio.

**G. B. Hamilton, Jr.**, a recent graduate from the Case Institute of Technology, is with the General Electric Co. in Schenectady, N. Y. He is a foundry engineer in the steel foundry of the Schenectady works.

**Gene W. Hinds** is a metallurgical engineer with the Geneva Steel Mill in Provo, Utah.

**Joseph H. Hoage** married Margaret Gordon in Denver on Oct. 23. They will make their home at 1314 Stevens Drive, Richland, Wash. He is with G. E. in Richmond.

**John J. Howard**, who retired from E. J. Lavino & Co. last year, can be reached at Garden Court, 47th and Pine Sts., Philadelphia 43.

**George J. Jameson** has transferred from the Technical Group, Naval Supply Depot, Clearfield, Ogden, Utah, to the Inspection Division, Seattle Supply Center, Bureau of Federal Supply. His primary duty is strategic and critical materials inspection.

**Percy E. Landolt**, consulting engineer of New York City, has been reelected president of the Association of Consulting Chemists and Chemical Engineers.



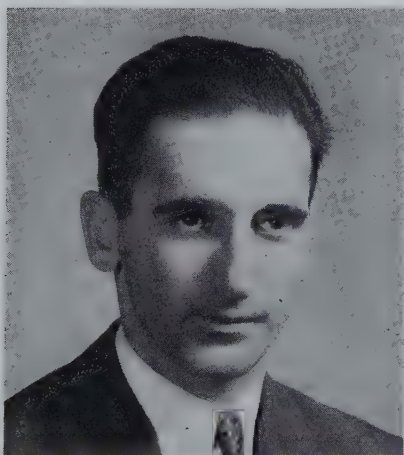
**Robert E. Lenhart** is working for the General Electric Co. His address is 6 Union St., Schenectady, N. Y.

**R. J. Loree** is associated with Sherritt Gordon Mines Ltd. at their pilot plant in Ottawa, Ont.

**Robert N. Lynch** became sales manager of the heat treating materials division of the Park Chemical Co., Detroit, on Oct. 1. With the company for ten years, he had been working as metallurgical sales and service engineer in the Michigan-Ohio territory.

**M. Merlub-Sobel** has been appointed associate professor of chemical engineering and metallurgy at the Hebrew Institute of Technology, Haifa, Israel.

**Jordan S. Murray** is a junior process engineer with the U. S. Potash Co. His address is 407 S. Lake St., Carlsbad, N. Mex.



Bernard S. Lement

**Bernard S. Lement**, formerly on the research staff at MIT assigned to a project on the dimensional stability of metals, received his Sc.D. degree in metallurgy there, and is now assistant professor of metallurgy at the University of Notre Dame.

**V. A. Phillips** has returned to England after completing graduate studies at the Hammond Metallurgical Lab., Yale, and is a research student at the Cavendish Lab., Cambridge, England.

**Maurice Sadowsky, Jr.**, is in the employment of the Inland Steel Co. at Gary, Ind., as a metallurgical trainee.

**James Howell Smith**, formerly in the research department of the Metals Refining Co., division of the Glidden Co., Hammond, Ind., has transferred to New York City as sales representative of Metals Refining in the Eastern states. His headquarters are at Rm. 802, 52 Vanderbilt Ave. Recently Mr.

Smith was commissioned a Captain in the Chemical Corps of the U. S. Army Reserve.



John P. Spielman

**John P. Spielman**, formerly professor of metallurgy at the Montana School of Mines, has been appointed dean of the School of Mines at the State College of Washington. His address is 1619 Fisk St., Pullman, Wash.

**H. T. Sumsion**, previously with the University of Utah, is now senior research engineer in the research department of the Carborundum Co., Niagara Falls, N. Y.

**Samir Tahir** has become metallurgist in the chemical department of the Egyptian Government. His address is 22 Dar El Shefa St., Garden City, Cairo.

**Lars H. Villner** is a research engineer for Jernkontoret in Stockholm.

**Theo J. Voll** recently became metallurgist for the Keystone Carbon Co. His address is Box 146A, Rt. 1, Saint Marys, Pa.

### —In Petroleum Circles—

**Phillip N. Bailey**, formerly with the Union Oil Co. of California, is taking graduate work at the University of Tulsa.

**Douglas Ball** went on leave of absence from the Phillips Petroleum Co. in July to become assistant to Max Ball, oil and gas consultant, in Washington. He is also attending classes at George Washington University. Mr. Ball recommends observance of certain portions of the fauna of downtown Washington at lunchtime for all engineers and geologists, especially on breezy days.

**John R. Brack** is employed by the Ohio Oil Co. as a trainee at the Yates field in Iraan, Texas. He gets his mail at Box 523, Iraan.

**Joe Chastain** is engineer representative for the Bethlehem Supply Co., Midland, Texas.

**Lester D. Collier** is a junior geologist with the Union Oil Co. of California at Shreveport, La.

**J. E. Eckel** resigned from the Oil Center Tool Co. on Oct. 15. His address is 300 Carson Court, Houston 4.

**Joy Elledge** is progress engineer for the Transcontinental Gas Pipe Line Corp. Mail goes to his home at 1002 Annex, Dallas.

**Earl Ellis** has a job as geophysicist with the Humble Oil and Refining Co. His mail goes to Box 212, Franklin, La.

**F. A. Graser** has returned to the ranks of petroleum consultants with his office at his home 421 Oak Lane, San Gabriel, Calif., after a year with the Cleveland Oil Co. in Los Angeles.



Harvey T. Kennedy

**Harvey T. Kennedy** has joined the staff of the petroleum engineering department of Texas A&M as distinguished professor of petroleum engineering. He had been with the Gulf Research and Development Co. in Pittsburgh.

**Lloyd A. Hamann** graduated from the University of Southern California last June. He is employed by the Stanolind Oil and Gas Co., working as an engineer trainee in the Slaughter area near Brownfield, Texas.

**R. D. Horst** is working as an engineer trainee with the exploitation department of the Shell Oil Co. At present his assignment is at the Weeks Island field, La.

**Harry L. Horton** has become associated with Dowell Incorporated as a junior engineer.



**John B. Hundley, Jr.**, went to work with the Richfield Oil Corp. last April. He is working in the production department at Cuyama Valley, Calif.



V. Dale Martin

**V. Dale Martin** resigned as head of the land department for the Venezuelan Atlantic Refining Co. in Caracas, to return to the States, where he is engaged in the promotion of certain oil properties for his own account. His address is 326 N. Minnesota Ave., Columbus, Kans.

**E. C. Jacobsen** is a geologist for the Standard Oil Co. of California. He is working in the Los Angeles Basin and living at 632 Longfellow, Hermosa Beach, Calif.

**A. A. McArthur** is a reservoir engineer trainee with The Texas Co. His mail goes to Box 827, El Campo, Texas.

**Paul Meadows** graduated from Texas Tech last January and is working for the Bureau of Mines petroleum experiment station at Bartlesville, Okla., as a petroleum engineer.

**Edward B. Kuhne** is an engineer revenue agent with the Bureau of Internal Revenue, stationed in New Orleans. He was with the Halliburton Oil Well Cementing Co.

**Joe A. Laird** is a member of the faculty of the department of petroleum engineering at Texas A&M College, College Station.

**Howard C. Lawton** has recently entered the consulting business and has set up the Lawton Research Laboratory at Fillmore, Calif. He will study problems on rotary drilling fluids, well reconditioning methods and analyses.

**William A. Libby** works as a guide for the National Park Service at Carlsbad Caverns in New Mexico. He

graduated from Texas College of Mines last May.

**Theodore A. Link**, consulting geologist of Toronto and Calgary, and vice-president of the AAPG and GAC, has been giving a series of lectures sponsored by the AAPG. He has visited Vancouver, Los Angeles as well as parts of West Texas and New Mexico.

**Carl Lund** has the post of sales and advertising manager with the Loeffler-Greene Supply Co. in Oklahoma City, Okla.

**Edgar N. Mills** has the job of junior engineer with the Pan American Production Co. His mail is sent to Box 241, Silsbee, Texas.

**T. Noordhuis** is in The Hague, Netherlands, as production engineer for Bataafsche Petroleum Maatschappij.

**Joseph E. Pogue**, head of the Chase National Bank's petroleum department, will relinquish his position as a Chase vice-president at the end of the year, but will retain his close affiliation with the bank in a consulting and advisory capacity on a part-time basis. He will continue his present office at 18 Pine St., New York City.



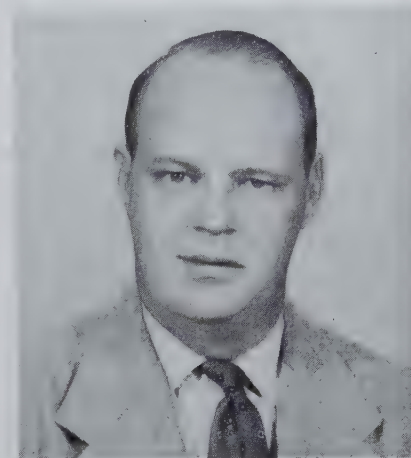
V. J. (Mike) Mercier

**V. J. (Mike) Mercier** has been made sales manager for the Mountain Iron and Supply Co., Wichita, Kans. Formerly with the Lane-Wells Co., he is well known in the Mid-Continent for his many papers on well logging and geophysics.

**Marvin A. Remke** was transferred in August to the Phillips Petroleum Company's West Texas district as staff engineer on operational problems. Now strictly in oil territory, he lives at 302 N. Kelley, Odessa, Texas.

**Clovis E. Rodelandier** is roustabout engineer for the Stanolind Oil and Gas Co., Box F., Hobbs, N. Mex.

**Robert L. Richardson** graduated from USC in June of 1948 and that August went to work for the Rocky Mountain Drilling. After five months on the rig floor he went into the main office as a junior engineer. This April he and Betty McCall were married.



Robert G. Hill

**Robert G. Hill** recently resigned as assistant to the district superintendent of the Texas Petroleum Co. in Venezuela, to do graduate work in the department of mineral economics at Penn State.

**F. L. Schenck** has left the Atlantic Refining Co. to go with Oil Properties Consultants of Pasadena, Calif., as a geologist.

**Albert G. Sledge, Jr.**, joined the production department of the Skelly Oil Co. at Velma, Okla., last June as a petroleum engineer and in October was transferred to Carthage, Texas.

**Gordon T. Swanby** is a geophysicist with the Stanolind Oil and Gas Co. at Craig, Colo.

**Scott L. Taliaferro** has left the Stanolind Oil and Gas Co. His present work is that of consulting engineer in the partnership of Taliaferro and Slicker, 203 Judia Bldg., Cisco, Texas.

**L. K. Van Dongen** is a consulting engineer with Arthur G. McKee & Co. He receives mail at 115 W. 69th St., New York City 23.

**William R. Waag** is a training petroleum engineer with the Sohio Petroleum Co., working on production and drilling in the Illinois district.

**H. Edward Wendt** graduated from Texas A&M in June with degrees in both petroleum and mechanical engineering. That same month he went to work for the Tidewater Associated Oil Co. in the Hobbs, N. Mex., district.



## Obituaries

### Army Adams

AN APPRECIATION BY JACK M. EHRHORN

Army Adams died of wounds of a gun accident on the rifle range at Grass Valley, Calif., on Aug. 20, 1949. He had been practicing his marksmanship in anticipation of the coming deer season.

Army was born in Montrose, Colo., on July 27, 1884. His father, John Adams, was a West Point Cadet who served as a member of the West Point Military Academy Escort which accompanied President Lincoln to the White House. The unusual name of Army had this origin. Army attended Montana University School of Mines where he studied mining engineering. Army Adams married Miss Alice Gregory in Colorado in 1910. His wife and three sons survive him.

Army Adams spent a full life in the West and acquired a wide and varied experience in mining, milling, and construction. He had worked for the Liberty Bell Gold Mining and Milling Co., the old Taylor Foundry and Machine Works, United Comstock, Pacific Gas and Electric Co., Mazapil Copper, Deep Creek Placer, Porcupine Mining Co., and the Idaho-Maryland Mines Corp. In 1940 he joined the Poverty Hill Partnership on which operations were suspended in 1942 by Order L-208.

Army Adams had a fund of stories and was one of the most entertaining of men. His resourcefulness expressed itself in his interesting recounting of his experiences. Early in his career he wrote several western stories which were published. Army Adams could be as rough and tough as men come or he could be as gentle as a lamb. He was a keen student of human nature and became an expert judge of men. Men liked to work with Army; though he was a severe taskmaster, they knew him to be fair and just. He was a man of highest mental and moral integrity and he will ever serve as an inspiration to those who knew and loved him.

### Necrology

Date Elected	Name	Date of Death
1928	Walter B. Congdon	Oct. 20, 1949
1937	W. J. Felber	Mar. 18, 1949
1923	S. R. Guggenheim	Nov. 3, 1949
1926	Thomas K. Knox	Mar. 7, 1949
1924	Frank E. Lee	Oct. 19, 1949
1944	C. Hubert Lenhart	Oct. 18, 1949
1901	Archibald Little	June. 24, 1948
1897	John W. Mercer	Oct. 18, 1949
1948	Earl H. Moffett	Unknown

**Arno S. Winther** (Member 1914), former general manager of the Miami Copper Co., is dead. His eminent leadership, friendship, and wise counsel were enjoyed and appreciated by all mining men, from the highest to the lowest, with whom he came in con-



Arno S. Winther

tact. Mr. Winther studied mining engineering at the University of Minnesota and first put his education to work at the Silver King Mining Co. In 1904 he was working for Cerro de Pasco, returning to Utah six years later with the Utah Consolidated Mining Co. He was with United Comstock, California Zinc, Rawley Mines, and Anna Beaver Mines, before taking off for Africa and the N'Kana Mine. By 1936 he was back in the States with Miami Copper.

**Zar T. Crittenden** (Member 1914), plant metallurgist with the Pontiac Motor Car Division of General Motors, died March 25 in Detroit. After getting his degree from the Montana State School of Mines in 1913 he went to sampling and assaying for the Phillipsburg Mining Co. From Montana he moved on to the Beaver Lake mining district of Saskatchewan and then to Manitoba to work for the Flin Flon Exploration Co. By 1919 he was south of the border again, associated with the Oakland Motor Car Co. in Pontiac, Mich., as assistant metallurgist. By 1936 he was plant metallurgist of the Pontiac Motor Car Division of GM.

**Herbert Chester Greer** (Member 1903), president of the Preston County Coke Co., Cascade, W. Va., died in August 1948. Upon graduation from MIT he worked with the Tennessee Coal and Iron Co. as a steel melter in the open-hearth steel department. In 1903 he was manager of the coal and coke department of La Belle Iron Works in Steubenville, Ohio. He owned the Greer Limestone Co. in addition to his work as president of Preston County Coke Co.

**Harry Davenport** (Member 1946), superintendent of mines for the Warren Foundry & Pipe Co., Wharton, N. J., is dead. Mr. Davenport was born in Milton, N. J., in 1889, and lived and worked in that state all his life. At 24 he was superintendent of milling and concentration at the Mt. Hope Mines and became superintendent of mines of the Warren Foundry in 1942.

**James Dyer Jones** (Member 1917), vice-president in charge of engineering for H. A. Brassert & Co., New York City, is dead. Mr. Jones started life in Pittsburgh in 1881, filled his first job there. In 1907 he went to Duluth to work for the Minnesota Steel Co., was with the M. H. Treadwell Co., and then moved to Sault Ste. Marie as assistant chief engineer of the Algoma Steel Co., eventually becoming vice-president and general manager. In 1935 he was in Youngstown, Ohio, as chief engineer for Youngstown Sheet and Tube.

**John Henry Gill Monypenny** (Member 1930), metallurgist for Brown, Bayley's Steel Works, Sheffield, England, died early this year. Mr. Monypenny was born at Sheffield in 1885, went to University College there and had worked for Brown, Bayley's since his graduation in 1904. He was the author of "Stainless Iron and Steel," published in 1926, as well as various technical papers.

**Sidney Lavern Palmer** (Member 1921), metallurgist for the Federated Metals Division of American Smelting and Refining, died May 9. He began his association with American Smelting in 1915, doing engineering work along metallurgical lines at the various smelters and coal mines of the Company in Colorado. Three years later he was in charge of all engineering work and mechanical operations at the Arkansas Valley plant. Since then he had worked for the Company in Colorado and California. Mr. Palmer was born in Ottawa, Ill., in 1883 and received his B.S. degree from Kansas State. For five years he was principal of the high school at Hutchinson, Kans., and then joined AS&R. He developed a number of new metallurgical processes and was instrumental in obtaining patents on these processes, one of which was equipment for casting a new type of bar solder which is sold under the trademark castomatic. During the last several years he had been working on the development of wire solder at the Newark plant.

**Robert Covert Patterson** (Member 1928), who had worked with both the U. S. Bureau of Mines and the U. S. Geological Survey, died April 3 in Glendale, Calif. He had been supervisor of oil and gas operations of the



Pacific division of the USGS and deputy supervisor of oil and gas operations in the Rocky Mountain division of the USBM. Mr. Patterson was born in 1887 in Neville Island, Pa., and was graduated from Occidental College. He had worked for the Standard Oil Co. of California and the California State Mining Bureau.

**Rudolph Porter** (Member 1916), former manager of the Federal plant of the American Smelting and Refining Co. at Alton, Ill., died on July 10. He was born at Cincinnati, Ohio, in 1873. He started work for AS&R at the Pueblo plant and then went to Federal, Ill., as manager of the Federal Lead Co.

## MICHIGAN

**Detroit**—BENS, FREDERICK PETER. (C/S—J-M).  
**Ironwood**—HOWBERT, JOHN CHARLES. (C/S—S-J).  
**Ishpeming**—FISHER, WARREN EDWARD. (C/S—J-M). SUMNER, JOHN STEWART. (C/S—S-J).  
**Wakefield**—WALKER, THOMAS. (C/S—S-J).

## MINNESOTA

**Duluth**—WINN, GEORGE S. (M).  
**Hayfield**—LIVINGSTON, JEAN BURTON. (C/S—S-J).  
**Hibbing**—ALTHOUSE, JOHN GIVENS. (J). SIMON, ROBERT EDWARD. (C/S—S-J). WHALEY, HENRY PFEFFLE. (C/S—S-J).  
**St. James**—GROSS, JOSEPH RAYMOND. (C/S—S-J).

## MISSOURI

**St. Louis**—FIELDS, LESTER E. (J).

## MONTANA

**Butte**—BROWN, ALAN DAVID. (C/S—S-J). GRUNENFELDER, J. GEORGE. (R, C/S—J-M). ROBERTSON, FORBES SMITH. (C/S—J-M). RYAN, MARGARET ANN. (C/S—S-J).  
**Helena**—BAARSON, ROBERT EUGENE. (C/S—S-J).  
**Helmville**—STOUT, KOEHLER SHERIDAN. (C/S—S-J).

## NEVADA

**Reno**—GRUTT, EUGENE WADSWORTH, JR., (C/S—J-M). HORTON, ROBERT C. (C/S—S-J).  
**Ruth**—BAKEWELL, ALAN AVERIL, JR. (C/S—J-M).

## NEW HAMPSHIRE

**Center Harbor**—DAVISSON, ALAN TUTTLE. (C/S—S-J).

## NEW JERSEY

**Jersey City**—THOMPSON, RICHARD HULL. (C/S—S-J).  
**Matawan**—VOGEL, FERDINAND LINCOLN, JR. (C/S—S-J).  
**Metuchen**—CROCKETT, JOHN MORTON. (C/S—J-M).  
**Phillipsburg**—MORRISON, GEORGE WILBUR. (M).  
**Ridgewood**—THORNLEY, EDWARD WILLIAMS. (AM).  
**Tenafly**—VAUGHAN, ROYAL WORTH. (AM).  
**West Englewood**—HARSHBERGER. ROBERT PERRINE. (M).  
**Westfield**—ALLEN, FORREST EUGENE. (M).

## NEW MEXICO

**Hobbs**—RANDOLPH, WILLIAM. (C/S—S-J).  
**Jalisco**—BARRETT, JEAN MARTIN. (C/S—S-J).  
**Silver City**—DESVAUX, JACK V. (R, C/S—S-M).  
**Socorro**—SMITH, CLAY TAYLOR. (C/S—J-M).

## NEW YORK

**Baldwin**—CARTER, CALVIN B. (M).  
**Bayside**—ROSE, FRED DAVID. (C/S—S-J).  
**Bronxville**—BRICE, JOHN W. (M).  
**Brooklyn**—MERRITT, PHILLIP LEONIDAS. (M). RUZICKA, JOHN. (C/S—J-M).  
**Crestwood**—TOWER, HORACE LINWOOD. (M).  
**Flushing**—BRUNSTETTER, DONALD RAY. (J).  
**Forest Hills**—CAYARD, PAUL CHARLES. (AM).  
**Garden City**—LEE W. J. (AM).  
**Glen Head**—RISING ALBERT EDWARD, JR. (C/S—J-M).  
**Great Neck**—JEFFREY, FREDERICK R. (AM).  
**New York**—ALLAN, ROY CARSON. (M). DAMP, RALPH E. (M). GOLSON, GEORGE ALBERT. (C/S—J-M). MACAUL, ROBERT THOMAS. (C/S—S-J). OVERBECK, ROBERT STEVENS. (C/S—J-M). PALTY, ADOLPH EDWARD. (J). STRAUS, OSCAR S. (AM).

# Proposed for Membership

Total AIME membership on Oct. 31, 1949, was 16,030; in addition 4413 Student Associates were enrolled.

## ADMISSIONS COMMITTEE

James L. Head, Chairman; Albert J. Phillips, Vice-Chairman; George B. Corless, T. B. Counselman, Ivan A. Given, Robert L. Hallett, Richard D. Mollison, and John Sherman.  
Institute members are urged to review this list as soon as the issue is received and immediately

to wire the Secretary's Office, night message collect, if objection is offered to the admission of any applicant. Details of the objection should follow by air mail. The Institute desires to extend its privileges to every person to whom it can be of service but does not desire to admit persons unless they are qualified.

In the following list C/S means change of status; R, reinstatement; M, Member; J, Junior Member; A, Associate Member; S, Student Associate; F, Junior Foreign Affiliate.

## ARIZONA

**Ajo**—RHOADES, RICHARD EVERETT. (C/S—S-J). RICKARD, FORREST ROBERT. (C/S—J-M).  
**Phoenix**—RODRIGUEZ, ERNEST R. (C/S—J-M).  
**TUNNELL**, HERBERT R., JR. (C/S—J-M).

## ARKANSAS

**Magnolia**—CORBY, WILLIAM ROBERT. (C/S—S-J). TURNER, VERNON JAY. (R, C/S—S-M).  
**Mountain Home**—WHITLA, RAYMOND E. (C/S—J-M).

## CALIFORNIA

**Alhambra**—LAIRD, EDWARD ARTHUR. (C/S—S-J).  
**Bellflower**—MORROW, THOMAS B. (C/S—J-AM).  
**Berkeley**—CHESTERMAN, CHARLES W. (M). WATSON, WILLIAM IRION. (C/S—J-M).  
**Hermosa Beach**—LOW, CEDRIC HERBERT. (AM).  
**La Jolla**—WARE, JOHN ALLEN, JR. (C/S—S-J).  
**Long Beach**—NISSEN, HAROLD EMIL. (C/S—J-M). WADE, FRANKLIN RUSSELL. (C/S—J-M).  
**Los Angeles**—ALPERT, LOUIS DAVID. (C/S—J-AM). ANDERSON, ROBERT EDWIN. (C/S—J-M). CHAMBERS, LAWRENCE SHINER. (M) EASTIN, BERTRAND PAUL. (M). HOCHMANN, LOUIS S. (C/S—S-J). KAESS, JAMES DONALD. (R, C/S—S-M). LIEDHOLM, CLARENCE CARLTON. (C/S—J-M).  
**Placentia**—KLOTZ, JAMES ALLEN. (J).  
**Sacramento**—SKEEHAN, STANLEY S. (C/S—J-AM).  
**San Bruno**—DUNCAN, WILLIAM ROY. (C/S—S-J).  
**South Gate**—WEBB, BYRON KENDALL. (R, C/S—J-M).  
**South Pasadena**—RICHARDSON, ROBERT LLOYD. (C/S—S-J).  
**South San Francisco**—ENDERLIN, CALVIN CHARLES. (C/S—S-J).  
**Torrance**—FORKUM, CARL (M).

## COLORADO

**Broomfield**—PARKER, CHARLES OWEN. (M).  
**Colorado Springs**—PEARL, RICHARD MAXWELL. (C/S—S-M).  
**Craig**—RHEA, JOHN WILLIAM, JR. (R, C/S—S-M).  
**Denver**—LINK, GEORGE HICKS. (J).  
**Lendville**—NEWLOVE, MARLIN EDGAR. (R, C/S—J-M).  
**Rangely**—WANNER, JOHN JAY. (J).

## CONNECTICUT

**Norwalk**—LYSOBEY, WILLIAM ROBERT. (AM).  
**South Glastonbury**—TAFT, BARRETT LORADO. (C/S—J-M).

## DELAWARE

**Wilmington**—EMERY, BYRON MATHEW. (C/S—S-J). RADTKE, SCHRADER FRED. (M).

## FLORIDA

**Mulberry**—HEWETT, LOUIS FRANKLIN. (M).  
**St. Augustine**—LITTLE, ARCHIBALD. (R—M).

## IDAHO

**Shelley**—STANIN, S. ANTHONY. (C/S—S-J).

## ILLINOIS

**Aurora**—GROLL, KENNETH REUBEN. (C/S—J-M).  
**Chicago**—AUBUCHON, EDWARD LEROY. (C/S—S-J). CORDES, JOHN ALLEN, JR. (C/S—S-J). FRIEDMAN, ISAAC W. (C/S—J-M). JOHNSON, STANLEY FRANCIS. (C/S—S-J). LEVER, EDWARD CARL. (C/S—S-J). UTTERBACK, GENE HYATT. (M).  
**Danville**—ALPERS, ELBERT EDMUND. (C/S—S-J).  
**Elgin**—McGAUGHEY, RAYMOND LEO. (C/S—S-J).  
**Hillsboro**—WAMPLER, HAROLD RAY. (C/S—J-M).  
**Lake Forest**—INTO, A. NORMAN. (M).  
**Robinson**—HOELSCHER, JAMES W. (C/S—S-J).  
**Washington**—OBERLE, THEODORE LORING. (C/S—J-M).

## INDIANA

**Bloomington**—DEISS, CHARLES FREDERICK. (M).  
**Gary**—PURCELL, STEPHEN McCABE. (C/S—J-M).  
**Notre Dame**—LEMENT, BERNARD SAMUEL. (C/S—J-AM).  
**Valparaiso**—BOEHME, HAROLD CLAYTON. (J).

## IOWA

**Keokuk**—OTHMER, LESLIE EARL. (R, C/S—AM-M).

## KANSAS

**Chase**—AARING, FLOYD DANIEL. (C/S—S-J).  
**Ellis**—RENNING, JOHN ALFRED. (C/S—J-M).

## LOUISIANA

**Monroe**—LEE, JOSEPH L. (M).  
**New Orleans**—CREWS, ROBERT NELSON. (AM).  
**Shreveport**—BEST, WILLIAM CLAYTON. (C/S—J-M). McNEIL, LAURENCE ALEXANDER. (J). PATTERSON, BENJAMIN FRANKLIN, JR. (C/S—J-M).  
**Vinton**—DAWSON, ARCH. (M).  
**Vivian**—ELLIS, WILLIAM FREDERICK. (C/S—S-J).

## MARYLAND

**Silver Spring**—HOPKINS, GEORGE RANDOLPH. (M).

## MASSACHUSETTS

**Cambridge**—BECKER, J. J. (J).  
**Melrose**—BRAHAM, MARTIN BERNARD. (C/S—J-M).



**Schenectady**—HOWE, JOHN PERRY. (M). NISBET, JAMES DOUGLAS. (AM). PUGH, JOHN WILLIAM. (C/S—S-J). TURNBULL, DAVID. (AM). **Star Lake**—CRUMP, ROBERT M. (R, C/S—J-M). **Suffern**—BARRON, WILLIAM MARSHALL. (C/S—S-J). **Troy**—EBELING, DOLPH GEORGE PHILIPPI. (M). **White Plains**—KOPECKI, ERNEST STEPHEN. (C/S—J-M).

#### NORTH CAROLINA

**Asheville**—VAN HORN, EARL CARLTON. (C/S—J-M).

#### OHIO

**Barnesville**—GRIMSLEY, JOHN DONALD. (C/S—S-J). **Columbus**—BAKLANOFF, NICOLAS WLADIMIR. (M). **Dayton**—SCHNEBLE, A. WILLIAM. (C/S—J-M). **Mansfield**—SANFORD, JUSTIN H. (AM). **Zanesville**—CHILDRESS, ALBERT ALISON. (M). COULSON, FRANK. (M). LINCOLN, RICHARD V. B. (M).

#### OKLAHOMA

**Bartlesville**—POWELL, JOHN PHILIP. (C/S—J-M). WILSON, CONNIE LEE. (J). **Duncan**—TAPPER WILFRED. (M). **Miami**—KRISHNASWAMY, RAMA-NATHA. (C/S—S-J). **Norman**—COBBS, JAMES HAROLD (C/S—S-J). **Oklahoma City**—BEEBE, BYRON WARREN (M). LOWE, ROBERT MONROE. (C/S—S-J). RALPH, RUSSELL GLENN. (M). **Tulsa**—HULINGS, NORMAN McDERMOTT, JR. (J). NIELSEN, EMERY H. (M). **Velma**—BOUSFIELD. JOHN CHANNING. (C/S—S-J). **Wewoka**—MASSAD, ALEXANDER HAMILTON. (R, C/S—S-J).

#### OREGON

**Mill City**—JENSEN, NORMAN CURTIS. (C/S—S-AM). **Port and**—DEMPSTER, REGINALD CHARLES. (M).

#### PENNSYLVANIA

**Bethlehem**—GUIDER, JAMES WALTER. (C/S—J-M). **Ellsworth**—BURNS, FRANK ALOYSIOUS. (C/S—S-J). **Forty Fort**—JAMES HERBERT MORRIS. (M). **Franklin**—MULOCK, RICHARD BUCKLES. (C/S—J-M). **Harrisburg**—STEPHENSON, ROBERT CHARLES. (C/S—J-M). **Hopwood**—SHOTT, ORVILLE ORA. (R, C/S—S-AM). **Kingston**—EDWARDS, MYLES WILLIAM. (M). **Johnstown**—CUNNINGHAM, WILLIAM ROY. (M). **Manor**—BEAMER, PAUL WILLIAM. (C/S—S-J). **Masontown**—GIROD, JAMES BERTHOLDE. (C/S—S-J). **New Brighton**—WEHN, JOHN GIRT. (M). **New Kensington**—LYLE, JOHN PAUL, JR. (R, C/S—S-J). **Pittsburgh**—FAIRLEY, ALBERT LANGLEY, JR. (C/S—J-M). HARMON, JOHN PAUL. (C/S—J-M). RATZ, GEORGE ALBERT. (J). RITTER, WAYNE G. (C/S—J-AM). **Portsville**—CRUM, STANLEY EDWARD. (M). **Scranton**—LEE, EDWARD JAMES. (C/S—S-J). **Sewickley**—BRASSERT, CHARLES ALEXANDER. (M). **Uniontown**—REED, FREDERICK JONES. (M). STACY, RICHARD HAROLD. (C/S—J-M). WERFT, AUGUST R. (M). **Vandergrift**—WILDE, RICHARD ARNOLD. (C/S—J-M). **West Brownsville**—KRESE, JAMES MARTIN. (C/S—J-M). **White Haven**—JENKINS, CARL S. (M).

#### SOUTH DAKOTA

**Belle Fourche**—MARSHALL, LAWRENCE KILBURN. (C/S—J-M). **Rapid City**—EVANS, OWEN DANFORTH. (C/S—J-M). TULLIS, EDWARD LANGDON. (C/S—J-M).

#### TENNESSEE

**Columbia**—GUENTHER, WILLIAM FREDERICK, JR. (C/S—J-M). **Jefferson City**—MILLER, HOWARD WILLIAM. (C/S—J-M). **Oak Ridge**—STEPHEN, HANS RICHARD. (C/S—J-M).

#### TEXAS

**Abilene**—REEVES, CARL LYNCH. (M). **Alice**—HOLLAND, TURNER BOYD. (M). **Alvin**—PIERCE, VINTON DOUGLAS. (C/S—S-J). **Amarillo**—SEIBEL, CLIFFORD WINSLOW. (M). **Austin**—PULS, WILLIAM LEROY. (R, C/S—S-J). **Beeville**—PRINCE, NOONAN ALBERT. (J). **Bellaire**—WILLIAMS, CHARLES EDWIN, JR. (AM). **Breckenridge**—REAUGH, ORLAND HARRY. (M). **Carthage**—SLEDGE, ALBERT G., JR. (C/S—S-J). **Corpus Christi**—CULWELL, WARREN DALE. (J). **Dallas**—HUTCHINSON, CHARLES ANGEVINE, JR. (J). KOCH, HOWARD ALEXANDER. (J). SHEPHERD, GEORGE FREDERICK. (R, C/S—J-M). STEPHENS, RICHARD GERRARD. (C/S—S-J). WARD, THEODORE GARLAND. (C/S—S-J). **El Campo**—AUBUCHON, PIERRE MARVIN. (J). McARTHUR, ARTHUR A. (C/S—S-J). **El Paso**—BELL, MARION S. (J). **Fort Worth**—TAPP, THEODORE LELAND. (M). **Hawkins**—McLAREN, CORNELIUS FORT, JR. (C/S—J-M). **Houston**—CLOUD, GOULD HENDERSON. (M). HAMEL, ROGER C., JR. (M). HARTUNG, GEORGE HERMAN. (M). HOLMAN, OSCAR KEITH. (C/S—J-M). LEWIS, RAY C. (M). LOBDELL, WALTER REGINALD. (M). LOCKWOOD, JAMES POTTER. (J). MATTHEWS, CHARLES SEDWICK. (J). ROOF, JACK GLYNDON. (M). **Kingsville**—SUMAN, RICHARD HARLAN. (C/S—S-J). **Levelland**—LANE, ROBERT DANIEL. (J). RODENBERGER, CHARLES ALVARD. (J). WADLINGTON, WILLIAM HAMILTON. (C/S—S-J). **Liberty**—PERILLOUX, CHARLES JOSEPH. (J). **Longview**—FULLER, C. CLARK, JR. (J). **Lubbock**—JOHNSON, PHILIP. (M). **Midland**—PATTON, MARK STEEVENS, JR. (C/S—J-M). **Odessa**—COCANOWER, ROBERT DUNLAVY. (M). **Sunny**—RABALAIS, MELFORD FRANCIS. (C/S—S-J). **Yoakum**—WAKE, HERBERT NEIL. (C/S—S-J).

#### UTAH

**Bingham**—ALLAN, CHARLES WALLACE. (C/S—S-J). **Cedar City**—COOK, KENNETH LORIMER. (C/S—J-M). **Lark**—BINGHAM, CLARENCE P. (R, C/S—S-J). **Midvale**—HINKS, WARREN HENRY, JR. (C/S—S-J). **Salt Lake City**—BARBER, CYRIL JAMES. (M). CAREY, GLEN ALBERT. (C/S—S-J). EVERETT, FLOYD DAVIS. (C/S—J-M). **Tooele**—TOWNSEND, BYRON E. (M).

#### VIRGINIA

**South Hill**—SUTTON, HAROLD LEE. (C/S—J-AM).

#### WASHINGTON

**Seattle**—BRUGMAN, JOSEPH JOHN. (C/S—J-M). STOLL, WALTER CLERIOUS. (C/S—J-M). **Spokane**—ZOLDOK, STEPHEN WILLIAM. (C/S—J-M). **Tacoma**—BUTLER, EARL BLUETT. (M). RICHARDSON, FREDERICK WILLIAM. (C/S—S-J).

#### WEST VIRGINIA

**Bluefield**—WALKER, WILLIAM WADE. (M). **Huntington**—HEBDEN, EDWARD H. (M). HASKELL, JAMES BLISS. (M). **Logan**—SWANGO, ARBERRY BERYL. (J). **Monaville**—LOPES, CARLOS JOSE. (M).

**Mount Hope**—MCINTIRE, SAMUEL THOMAS. (M). **Neills**—BARTONY, HENRY ERNEST (C/S—S-J). **Parkersburg**—JOHNSTON, HOWARD FEIGHNER. (C/S—J-M). **Pineville**—REDYARD, JESSE. (AM).

#### WISCONSIN

**Hales Corners**—SNAVELY, J. W. (M). **Milwaukee**—BRONKALA, WILLIAM JENNINGS. (C/S—S-J). **Shorewood**—HIGGINS, GEORGE W. (C/S—J-M).

#### WYOMING

**Casper**—RICHARDS, SIDNEY BRUCE. (C/S—J-M). SCHUMACHER, JOHN EARL, JR. (C/S—S-J). **Cody**—HAAS, GILBERT GEORGE (C/S—J-M). MORTON, LELAND MATTHEWS. (J). PATERSON, JOHN FRUIN. (C/S—J-M). **Grass Creek**—SPIELES, EARL LOYD. (C/S—S-J). LANDER, ROBERT BARTLETT. (J).

#### ALBERTA

**Calgary**—McPHERSON, WILLIAM JOHN. (C/S—S-M).

#### BRITISH COLUMBIA

**Allenby**—TAIT, ROBERT JOHN COMP-TON. (C/S—S-AM). **Vancouver**—GUNNING, HENRY C. (M).

#### ONTARIO

**Holtbyre**—CAYLOR, RUSSELL D. (C/S—J-M). **Madsen**—VAN LOON, LESLIE HART. (C/S—J-M). **Port Colborne**—CORDNER, T. (M).

#### GUERRERO

**Mexcala**—CROSBY, ALLEN FOSTER. (C/S—J-M).

#### QUERETARO

**Cadereyta**—FAULKNER, JAMES E. JR. (C/S—S-J).

#### NICARAGUA

**Managua**—BATH, KENNETH ARTHUR. (C/S—S-J).

#### BOLIVIA

**Oruro**—ANTEZANA, CARLOS JORGE. (C/S—S-AM).

#### BRAZIL

**Rio de Janeiro**—ODDONE, DECIO SAVERIO. (C/S—J-M).

#### CHILE

**Conagua**—SWAIN, ROBERT LOCHE. (C/S—S-J).

#### COLOMBIA

**Antioquia**—SCHOU, HANS HELGE. (C/S—J-M).

#### VENEZUELA

**Barcelona**—HUIE, JAMES POWELL. (AM). ROBERTS, CHARLES C. (C/S—J-M). WILSON, LEJEUNE. (C/S—S-J).

#### ENGLAND

**Cumberland**—YOUNG, JOHN D. (J).

#### FRANCE

**Paris**—CALLEYA, GEORGES XAVIER. (M). JACQUET, PIERRE ARMAND. (M). PETIT, DANIEL. (M).

#### SWEDEN

**Huskvarna**—KOUKKOLA, KALEVI VILJAM. (J).

#### TRANSSAAL

**Johannesburg**—ENSLIN, JOHANNES STEPHENUS. (C/S—S-M). MARTINCEVIC, NICHOLAS. (C/S—J-M).

#### FEDERATED MALAY STATES

**Kuala Lumpur**—EDMONDS, HAROLD JOSEPH. (C/S—AM-M).

#### INDIA

**Kolar Gold Field**—NARAYANA RAO, C. E. (C/S—J-AM). **Oorgaum**—SUBRAMANYAM, MATHURMUTHU. (C/S—J-M).

#### PHILIPPINE ISLANDS

**Surigao**—MERWIN, ROLAND WINFIELD. (M).



# Melting Points in the System $\text{TiO}_2\text{-CaO-MgO-Al}_2\text{O}_3$

H. SIGURDSON\* and S. S. COLE,\* Member AIME

## Introduction

The melting points of mixtures of titanium dioxide and other titanates have been reported to a limited extent as binary systems and some results have been reported in conjunction with silicon dioxide. The limited data indicated that a low melting zone might exist in a ternary or quaternary system of  $\text{CaO-MgO-TiO}_2\text{-Al}_2\text{O}_3$ , since the eutectics reported in the binary systems of  $\text{MgO-TiO}_2\text{-TiO}_2$ ,  $\text{Al}_2\text{O}_3\text{-TiO}_2\text{-TiO}_2$  and  $\text{CaO-TiO}_2\text{-TiO}_2$  were of about equivalent composition of  $\text{TiO}_2$ . The importance of a low melting region with a high titanium oxide value in the developing of high titanium slags is fully appreciated by metallurgists. A comprehensive study was undertaken to establish basic information on low melting titanate mixtures.

## Compounds in the System $\text{CaO-MgO-TiO}_2\text{-Al}_2\text{O}_3$

Preparation of the titanates of  $\text{CaO}$ ,  $\text{MgO}$  and  $\text{Al}_2\text{O}_3$  confirmed reported data that the following compounds could be formed under oxidizing conditions in solid state reactions:

$\text{CaO-TiO}_2$ ,  $2\text{MgO-TiO}_2$ ,  $\text{MgO-TiO}_2$ ,  
 $\text{MgO-2TiO}_2$  and  $\text{Al}_2\text{O}_3\text{-TiO}_2$ .

Under oxidizing conditions it was not possible to react  $\text{MgO}$  with  $\text{TiO}_2$  in mol ratios higher than 1:2 without having unreacted  $\text{TiO}_2$  in the product, nor was it possible to form calcium or aluminum titanates with a higher mol ratio of  $\text{CaO}$  and  $\text{Al}_2\text{O}_3$  to  $\text{TiO}_2$  than 1:1.

By fusing mixtures of  $\text{Al}_2\text{O}_3\text{-TiO}_2$  and  $\text{MgO-2TiO}_2$  together a series of solid solution products were obtained, which showed an  $\text{MgO-2TiO}_2$  X ray diffraction pattern shifted to smaller interplanar spacings.

## Range of Investigation

Data in the literature<sup>1</sup> indicated that these titanates had melting points which ranged from 1645 to 1860°C.

Since the purpose of the study was primarily to provide useful data for smelting titaniferous ores, the work was restricted to the zone of the system which would include the crystalline phases present in the slags. The explored limits were bounded by  $\text{CaO-TiO}_2\text{-MgO-TiO}_2\text{-TiO}_2$  in the base plane. The system was extended to a fourth component  $\text{Al}_2\text{O}_3\text{-TiO}_2$  since many titaniferous ores contain appreciable amounts of  $\text{Al}_2\text{O}_3$ .

In the quaternary system  $\text{CaO-TiO}_2\text{-MgO-TiO}_2\text{-TiO}_2\text{-Al}_2\text{O}_3\text{-TiO}_2$ , a tetrahedron was used to represent graphically the components, with one component at each point of the tetrahedron.

In the base plane, mixtures were pre-

pared to represent fairly uniform changes of composition over the desired range expressed on a mol percent basis.  $\text{Al}_2\text{O}_3\text{-TiO}_2$  was brought into the system in increments of 10 mol pct up to 40 pct. Mixtures were then made up for each of the  $\text{Al}_2\text{O}_3\text{-TiO}_2$  planes. The ranges employed were then represented by five planes cutting the tetrahedron at 10 mol pct  $\text{Al}_2\text{O}_3\text{-TiO}_2$  intervals.

## Choice of Equipment

Although it was necessary for all smelting work to be done in a strongly reducing atmosphere, it was decided that all melting point determinations should be done under oxidizing conditions. This decision was prompted by the fact that  $\text{TiO}_2$  reduced to lower oxides in a reducing atmosphere with consequent changes in melting points.

The use of a micropyrometer and a platinum strip furnace for work of this type is adequately described in the literature.<sup>2</sup> With modifications, equipment was selected which provided a fairly rapid method of determining melting points of refractory oxides.

The final assembly, which was used, consisted of a platinum strip furnace and a Leeds and Northrup disappearing filament optical pyrometer attached to a special telescope which magnified the sample about 20 diam.

The furnace assembly consisted of a platinum strip  $0.005 \times 0.3 \times 2.2$  in. mounted on brass posts on a refractory base. This was enclosed in a black steel shell which had a 2-in. opening at the top for purposes of sighting the telescope.

The platinum strip was in series with two 0.04 ohm nichrome resistors connected to the secondary of a 2 kw transformer which supplied 17 volts. The current to the primary was varied

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<sup>1</sup> References are at the end of the paper.



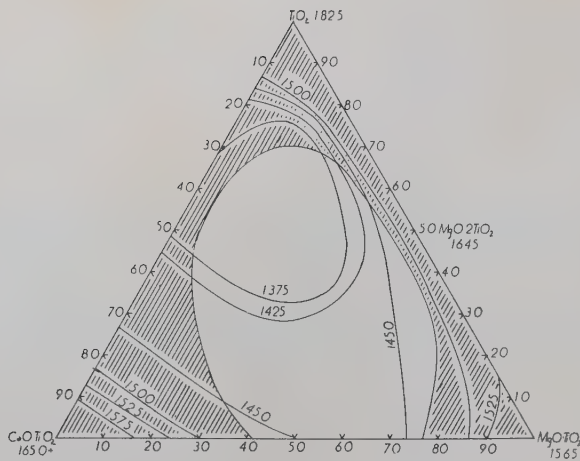


FIG 1—Melting equilibria for system  $\text{CaO} \cdot \text{TiO}_2$ - $\text{MgO} \cdot \text{TiO}_2$ - $\text{TiO}_2$ . Clear area-fluid zone. Cross-hatched area-viscous zone.

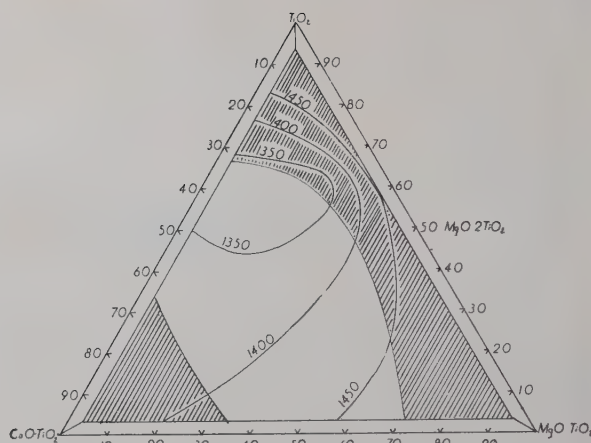


FIG 2—Melting equilibria for system  $\text{CaO} \cdot \text{TiO}_2$ - $\text{MgO} \cdot \text{TiO}_2$ - $\text{TiO}_2$ - $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  (10 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  layer). Clear area-fluid zone. Cross-hatched area-viscous zone.

by means of a 35 ohm variable resistor. Large increases of current in the system were made with this resistor. Very fine adjustments were made by means of the sliding contacts on the nichrome resistors.

## Preparations of Mixtures

The compounds  $\text{CaO} \cdot \text{TiO}_2$ ,  $\text{MgO} \cdot \text{TiO}_2$ ,  $\text{MgO} \cdot 2\text{TiO}_2$  and  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  were prepared from spectrographically pure  $\text{TiO}_2$ , and cp  $\text{CaCO}_3$ ,  $\text{MgO}$  and  $\text{Al}(\text{OH})_3$ . Stoichiometric amounts of the components were wet mixed, dried and pelletized. The pellets were then fired in platinum at  $1500^\circ\text{C}$  under oxidizing conditions for 3 hr to complete transformation to the compounds in each case. Chemical analyses of the compounds showed the materials to be on composition within 0.4 pct.

These compounds were then finely ground, wet-mixed in varying amounts to give the required compositions and sintered at  $1200^\circ\text{C}$  for at least 3 hr to give hard sintered products.

## Determination of Melting Points of Mixtures

Fragments of the sintered mixtures, which had been screened through a 100 mesh screen, were placed on the platinum strip and spread evenly to insure close contact between the platinum and the sample. The temperature of the strip was then raised rapidly to about  $1000^\circ\text{C}$ . Time was allowed for the sample to reach temperature stability and the temperature rise was then adjusted to about  $10^\circ\text{C}$  per min. until the melting point was reached.

The temperatures at which melting occurred were obtained by sighting the pyrometer on the platinum strip adjacent to the sample at right angles to the length of the strip and in line with the particles under observation. These precautions were necessary since the temperature gradient across the strip was quite steep.

The emissivity of the platinum strip in the open conditions which prevailed was considerably less than in black-body conditions so temperature corrections were applied in accordance with Table 16, p. 13, of Bureau of Standards "Pyrometric Practice" No. 170. The emissivity corrections for platinum were checked against the following substances whose melting points are reported in the literature:

Substance	Reported Melting Point $^\circ\text{C}$	Observed* Melting Point $^\circ\text{C}$	Deviation from Reported Value $^\circ\text{C}$
Gold	1063	1060	-3
$\text{Na}_2\text{Ti}_2\text{O}_7$	1128	1125	-3
$\text{BaF}_2$	1280	1270	-10
$\text{CaMgSi}_2\text{O}_6$	1391	1385	-6
$\text{MgTi}_2\text{O}_5$	1660	1645	-15

\* Emissivity corrections for platinum applied to observed temperature reading.

The resolving power of the telescope was sufficient to permit the observance in the fluid melts of the points at which the last solid particles became molten. In these melts the particles formed spheres very sharply at the melting point. The temperature of formation of spheres and the disappearance of the solid phase coincided. As a check, the temperature of the sample was lowered through the melting point to note the temperature of appearance of the solid phase. Good agreement was obtained.

In the range of melts which were

classified as viscous, it was difficult to determine accurately the point at which the particles became spherical, since they showed a rounding of the edges at the softening point and the transition to spheres was not nearly as sharp as in the mixtures which were fluid. Chief reliance was placed on the observance of the point at which the last observable solid particles disappeared.

Quenching of the samples did not assist in determining if the mixtures were completely molten, since the crystal growth of the titanates was so rapid that isotropic phases could not be obtained. Hence the actual determination of the points at which samples were completely molten rested very much with the judgment of the operator.

## Definition of Melting and Softening Points

The following definition of the melting point of a refractory material was used,<sup>2</sup> "The melting point of an irregularly shaped refractory particle is that temperature at which the viscosity (or crystalline rigidity) is overcome by the random motion of the molecules and at which the surface tension is sufficient to draw the particle into a globular shape."

In contrast to this, the "softening" point was considered to be "the temperature at which the corners of an angular piece of the refractory substance first became rounded." At this point there would be considerable unfused material present in the particle whereas at the melting point there would be only a trace of solid material in equilibrium with the fused portion.



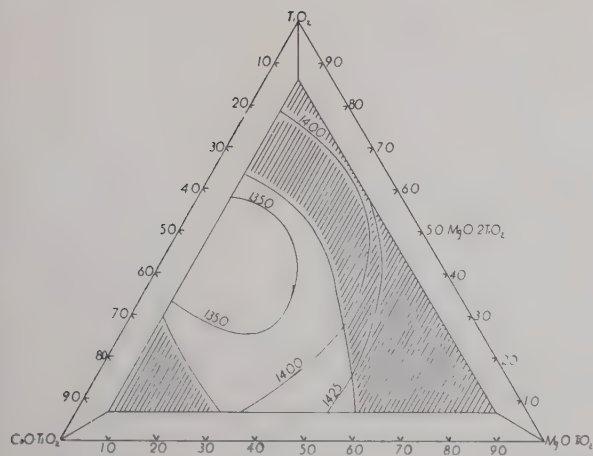


FIG 3—Melting equilibria for system  $\text{CaO-TiO}_2\text{-MgO-TiO}_2\text{-TiO}_2\text{-Al}_2\text{O}_3\text{-TiO}_2$ . (20 mol pct  $\text{Al}_2\text{O}_3\text{-TiO}_2$  layer.)  
Clear area-fluid zone. Cross-hatched area-viscous zone.

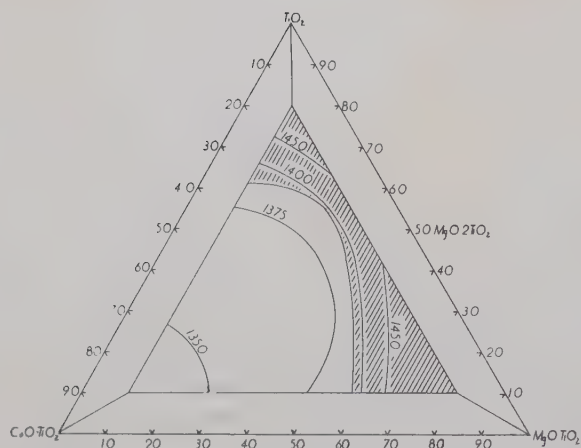


FIG 4—Melting equilibria for system  $\text{CaO-TiO}_2\text{-MgO-TiO}_2\text{-TiO}_2\text{-Al}_2\text{O}_3\text{-TiO}_2$ . (30 mol pct  $\text{Al}_2\text{O}_3\text{-TiO}_2$  layer.)  
Clear area-fluid zone. Cross-hatched area-viscous zone.

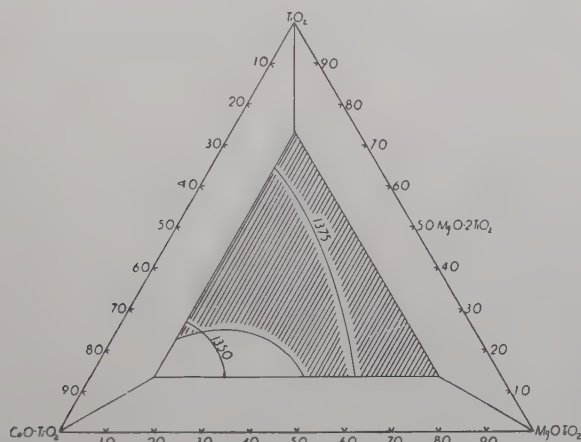


FIG 5—Melting equilibria for system  $\text{CaO-TiO}_2\text{-MgO-TiO}_2\text{-TiO}_2\text{-Al}_2\text{O}_3\text{-TiO}_2$ . (40 mol pct  $\text{Al}_2\text{O}_3\text{-TiO}_2$  layer.)  
Clear area-fluid zone. Cross-hatched area-viscous zone.

Table 1 . . . Patterns of  $\text{MgO} \cdot 2\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  and Intermediate Series of Mixed Crystals Spectrometer,  $\text{Cu K}\alpha, \lambda = 1.5374 \text{ \AA}$ , 2 rpm scan. No Corrections Applied

$\text{MgO} \cdot 2\text{TiO}_2$		80Mol % $\text{MgO} \cdot 2\text{TiO}_2$ 20Mol % $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$		60Mol % $\text{MgO} \cdot 2\text{TiO}_2$ 40Mol % $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$		40Mol % $\text{MgO} \cdot 2\text{TiO}_2$ 60Mol % $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$		20Mol % $\text{MgO} \cdot 2\text{TiO}_2$ * 80Mol % $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$		$\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$	
d/n $\text{\AA}$	I/I	d/n $\text{\AA}$	I/I	d/n $\text{\AA}$	I/I	d/n $\text{\AA}$	I/I	d/n $\text{\AA}$	I/I	d/n $\text{\AA}$	I/I
5.0	0.12	4.9	0.2								
4.9	0.12	4.8	0.4	4.8	0.3	4.8	0.4	4.8	0.7	4.71	0.47
3.51	1.00	3.44	1.00	3.43	0.7	3.40	1.00	3.39	1.0	3.35	1.00
2.86	0.09	2.83	0.08	2.82	0.05					2.65	0.7
2.75	0.6	2.72	1.00	2.71	1.00	2.70	0.7	2.68	0.5		
2.45	0.2	2.42	0.2	2.41	0.1	2.44	0.09			2.36	0.20
2.42	0.15	2.39	0.2	2.37	0.05	2.41	0.12				
2.22	0.15	2.21	0.2	2.19	0.15					2.14	0.31
2.19	0.2	2.17	0.25	2.16	0.1	2.16	0.2 <sup>w</sup>			2.11	0.30
1.967	0.3	1.951	0.3	1.943	0.25	1.931	0.4	1.91	0.2	1.897	0.52
1.876	0.6	1.850	0.6	1.836	0.25	1.822	0.3	1.81	0.4	1.792	0.44
1.846	0.15	1.836	0.1							1.687	0.28
1.754	0.15	1.738	0.1	1.725	0.1	1.713	0.15			1.606	0.27
1.665	0.15	1.653	0.2			1.629	0.09			1.578	0.28
1.634	0.2	1.621	0.2	1.613	0.1	1.601	0.1			1.519	0.06
1.551	0.3	1.532	0.2	1.524	0.15					1.484	0.36
1.535	0.2	1.528	0.2	1.514	0.1					1.449	0.08
1.424	0.09	1.409	0.1							1.371	0.14
1.378	0.08									1.305	0.16
1.358	0.15	1.343	0.1							1.266	0.11
1.318	0.08	1.283	0.09							1.253	0.11
1.265	0.10	1.256	0.1							1.219	0.09
1.245	0.10	1.235	0.07								

<sup>w</sup>—After relative intensity means wide line or lines.

\*—Very weak pattern.



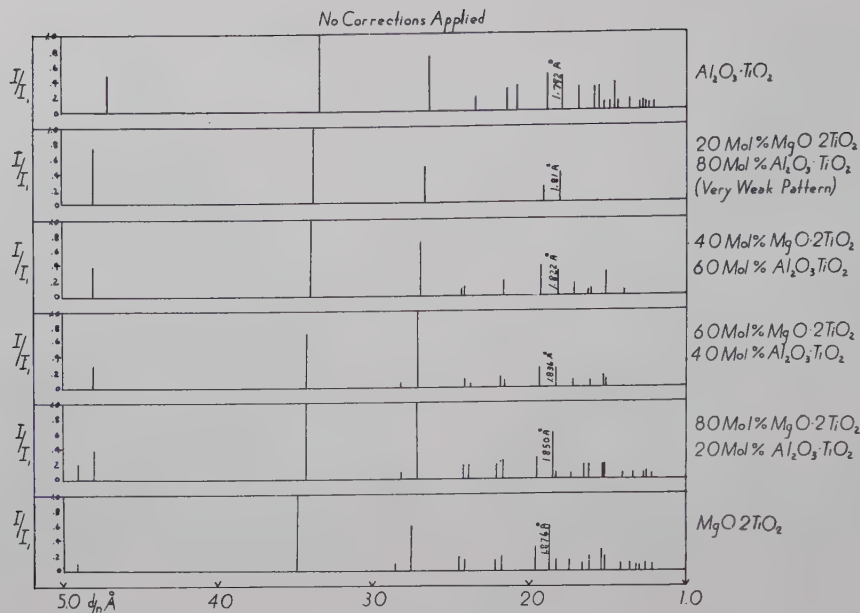


FIG 6—Patterns of  $\text{MgO} \cdot 2\text{TiO}_2$ - $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  and intermediate series of mixed crystals. Spectrometer,  $\text{Cu K}\alpha, \lambda = 1.5374 \text{ \AA}$ , 2 rpm scan.

## Differentiation between Fluid and Viscous Melts

Preliminary investigation of the melting points of mixtures of the titanates disclosed, in many cases, that if a sample were heated above the melting point, the surface tension was sufficient for several neighboring spheres to migrate into one mass.

If this occurred within  $100^\circ\text{C}$  of the observed melting point, the mixture was classified as "fluid." If this migration did not occur within this range the mixture was classified as "viscous."

## Phase Relationships

Equilibrium conditions prevailing at high temperatures were not obtained, since any possible transitions could not be prevented by the quenching method. No attempts were made to determine these equilibria by a method such as X ray diffraction studies of compounds at high temperatures.

In mixtures which had been fused and subsequently cooled to room temperature, the following compounds were observed:

1. In the base-plane  $\text{CaO} \cdot \text{TiO}_2$ - $\text{MgO} \cdot 2\text{TiO}_2$ - $\text{TiO}_2$  (See Fig 1).

(a) Below the  $\text{CaO} \cdot \text{TiO}_2$ - $\text{MgO} \cdot 2\text{TiO}_2$  tie-line,  $\text{CaO} \cdot \text{TiO}_2$ ,  $\text{MgO} \cdot \text{TiO}_2$  and  $\text{MgO} \cdot 2\text{TiO}_2$  were present in the proportions expected from the position of the mixture on the phase diagram.

(b) Above the  $\text{CaO} \cdot \text{TiO}_2$ - $\text{MgO} \cdot 2\text{TiO}_2$

tie-line,  $\text{CaO} \cdot \text{TiO}_2$ ,  $\text{MgO} \cdot 2\text{TiO}_2$  and  $\text{TiO}_2$  were present in expected proportions.

2. In the 10, 20, 30 and 40 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  layers of the System (See Fig 2, 3, 4 and 5).

Melts of the mixtures falling within these four planes crystallized to give  $\text{CaO} \cdot \text{TiO}_2$ ,  $\text{MgO} \cdot \text{TiO}_2$  and mixed crystals of  $\text{MgO} \cdot 2\text{TiO}_2$  and  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$ . X ray diffraction studies of mixtures of the series  $\text{MgO} \cdot 2\text{TiO}_2$ - $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$ , which had been fused and crystallized, showed that the two compounds form a continuous series of mixed crystals. The interplanar spacings derived from X ray powder diffraction patterns of representative members of the series are shown in Table 1 and Fig 6. Similar studies of mixtures falling within the above four planes, showed shifted patterns for  $\text{MgO} \cdot 2\text{TiO}_2$  or  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  depending upon the composition of the mixture. Uncombined  $\text{Al}_2\text{O}_3$  was never present in a melt of these mixtures.

## Conclusions

(1) The melting points of the various mixtures investigated are presented in Fig 1, 2, 3, 4 and 5. Mixtures which were classified as "fluid" in the molten state are within the clear areas of each figure. Shaded areas show the compositions which were classified as "viscous" in the molten state.

(2) In the base plane  $\text{CaO} \cdot \text{TiO}_2$ - $\text{MgO} \cdot \text{TiO}_2$ - $\text{TiO}_2$  a eutectic at  $1360^\circ\text{C}$  was established between  $\text{CaO} \cdot \text{TiO}_2$  and

$\text{MgO} \cdot 2\text{TiO}_2$  at 40 mol pct  $\text{CaO} \cdot \text{TiO}_2$  and 60 mol pct  $\text{MgO} \cdot 2\text{TiO}_2$ .

It was found, however, that melting points at about  $1375^\circ\text{C}$  were obtained with mixtures much higher in  $\text{TiO}_2$  than this combination (for example, a mixture of 13.5 mol pct  $\text{MgO} \cdot \text{TiO}_2$ , 20 mol pct  $\text{CaO} \cdot \text{TiO}_2$  and 65 mol pct  $\text{TiO}_2$  had a melting point of  $1375^\circ\text{C}$ ). This gave promise of the feasibility of working with fluid combinations much higher in  $\text{TiO}_2$  than would be encountered with  $\text{CaO}$  and  $\text{MgO}$  additions sufficient to place the resultant mixture on the tie-line between  $\text{CaO} \cdot \text{TiO}_2$  and  $\text{MgO} \cdot 2\text{TiO}_2$ .

3.  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  additions up to 30 mol pct did not adversely affect the melting temperatures of the mixtures, but a shift in the zone of fluidity toward the  $\text{CaO} \cdot \text{TiO}_2$  part of the system was evident.

40 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  additions to the system caused a sharp decrease in the zone of fluid melts and a 50 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  addition gave only viscous melts.

## References

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(c) H. V. Wartenburg, H. J. Reusch and E. Saran: System  $\text{CaO} \cdot \text{TiO}_2$ . *Ztsch. anorg. u. allgem. Chem.* (1936-37) **230**, 257.
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# Laboratory Smelting of Titaniferous Ores

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## Introduction

The smelting of titaniferous ores for the past hundred years has not been successful because of thickening of the slags in the furnaces. The interest in the utilization of these iron bearing materials has been due to the extensive deposits which have been found in various parts of the world.<sup>1</sup> The literature contains references<sup>2</sup> to the use of titaniferous ores in blast furnaces. None of these attempts has met with success due to the lack of sufficient information as to the cause of failures and to economic considerations. Evaluation of the published information, as to the probable cause of failure in various attempts, has indicated that the presence of lower valence titanium oxide and formation of titanium carbide contributed to the failure. It was apparent that severe reducing conditions which are present in a blast furnace had been one of the factors for such failures. The previous smelting operations had as a primary objective the recovery of iron from these ores. Under these conditions the highest recovery of iron could only occur with severe reducing conditions.

## Application of Melting Point Data

The application of the melting point data, from the system  $\text{CaO-MgO-Al}_2\text{O}_3\text{-TiO}_2$  reported in another paper,<sup>3</sup> to the smelting of titaniferous ores required an extrapolation of these melting points due to the presence of other impurities from the ores. Likewise, a study of the effect of varying  $\text{FeO}$  and reduced titanium content of the slag was necessary before the data could be fully applied. The melting point data under oxidizing conditions had clearly indicated that a fluid slag with a melting point in the temperature range of  $1370^\circ\text{C}$  could be obtained. The smelting of a titaniferous ore had as a

primary objective the production of a high titanium slag which was fluid and substantially free of iron and low in trivalent titanium. Since production of a high titanium slag was of primary importance, rather than the recovery of iron from these ores, the smelting concept could be altered to a marked degree. The ferrous oxide could be maintained at higher levels than is the normal practice in iron ores slags without being detrimental to the slags, although a loss in iron recovery resulted.

During the reduction of the molten mixtures of ferrous iron and titanium, carbon acts as the reductant for changing the valence of the titanium and iron. The reaction can go further than in the case of the reduction which occurs in iron-titanium solutions. The formation of metallic iron removes iron from the system, so that the titanium oxide can be reduced to trivalent and divalent titanium oxides and also form titanium carbide which removes the titanium from the slags as a soluble constituent. The problem, therefore, resolves itself into maintaining the proper amount of ferrous iron in the slag thereby avoiding the formation of appreciable amounts of trivalent titanium and no divalent titanium oxide or titanium carbide. The latter two have been found in the viscous

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<sup>1</sup> References are at the end of the paper.



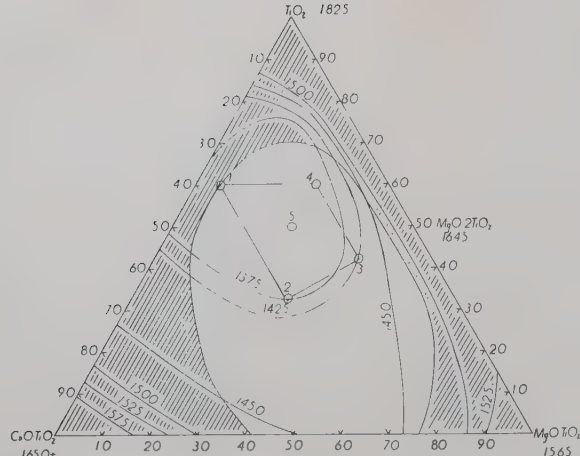


FIG 1—Melting equilibria for system  $\text{CaO} \cdot \text{TiO}_2$ — $\text{MgO} \cdot \text{TiO}_2$ — $\text{TiO}_2$ .

Clear area—Fluid zone  
Cross hatched area—Viscous zone  
Areas 1, 2, 3, 4—Normal slag zone

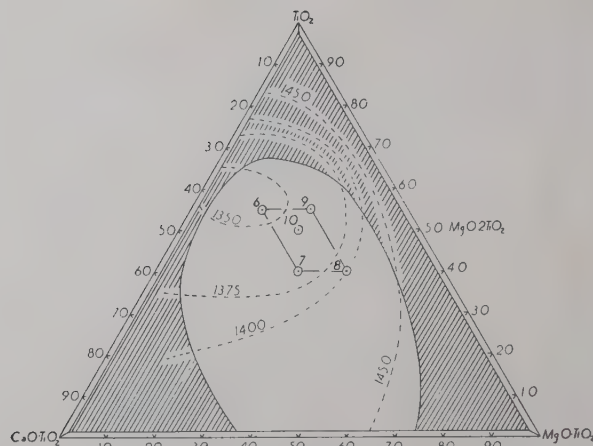


FIG 2—Melting equilibria for system  $\text{CaO} \cdot \text{TiO}_2$ — $\text{MgO} \cdot \text{TiO}_2$ — $\text{TiO}_2$ — $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  (5 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  layer) interpolated from Fig 1 and 3.

Clear area—Fluid zone  
Cross hatched area—Viscous zone  
Areas 6, 7, 8, 9—Normal slag zone

slags which have been made from the titaniferous ores in blast furnace operation. The avoidance of the formation of  $\text{Ti}_2\text{O}_3$  was also an important consideration since it is known that this oxide is homologous with  $\text{Al}_2\text{O}_3$ . It would be expected that the formation of  $\text{Ti}_2\text{O}_3$  would cause a viscous condition in a slag since this would be equivalent to addition of  $\text{Al}_2\text{O}_3$ . Likewise, the presence of reduced titanium in the slag was to be avoided since this was not desirable in the final solutions obtained from the digestion of the slag.

Most titaniferous ores contain as normal impurities small amounts of  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{CaO}$  and  $\text{SiO}_2$ . Since the four component system which had been studied did not include  $\text{SiO}_2$ , it was necessary in any preliminary calculations to assume that the  $\text{SiO}_2$  would function the same as  $\text{TiO}_2$ . This assumption was in slight error, but the characteristics of the slag produced were not appreciably affected. The titaniferous ores which were considered of primary importance in this study were the large ore bodies which are proven deposits in New York, Canada and Norway. Typical analyses of the ores used for the smelting are given in Table 1. The amount of  $\text{Al}_2\text{O}_3$  in these ores with reference to the  $\text{TiO}_2$  content varies slightly. The analyses have been calculated to show the  $\text{TiO}_2/\text{CaO}$ ,  $\text{TiO}_2/\text{MgO}$ , and  $\text{TiO}_2/\text{Al}_2\text{O}_3$  ratios. These ratios are the basic minima that would be obtained in a slag if no flux were to be used. The same information is utilized in considering the general composition that

Table 1 . . . Analysis of Titaniferous Ores

	MacIntyre Ilmenite Tahawus, N. Y. Pct	Baie St. Paul Ore Baie St. Paul, Quebec, Pct
$\text{TiO}_2$	44.6	38.8
$\text{FeO}$	36.7	28.3
$\text{Fe}_2\text{O}_3$	8.2	19.7
$\text{CaO}$	0.8	0.9
$\text{MgO}$	2.6	3.8
$\text{SiO}_2$	4.0	3.5
$\text{Al}_2\text{O}_3$	3.2	2.8
$\text{V}_2\text{O}_5$	0.04	0.29
$\text{Cr}_2\text{O}_3$	0.02	0.09
S	0.24	0.67
$\text{P}_2\text{O}_5$	0.045	0.04
$\text{TiO}_2/\text{CaO}$	55.7	43.0
$\text{TiO}_2/\text{MgO}$	17.2	10.2
$\text{TiO}_2/\text{Al}_2\text{O}_3$	13.9	13.8

should be adopted from melting point data. The ratio of  $\text{TiO}_2/\text{Al}_2\text{O}_3$  is the most critical in considering different ores with respect to general smelting and production of a fluid slag. This is particularly the case in ores which cannot be dressed to produce a low  $\text{Al}_2\text{O}_3$  content.

With this basic amount of  $\text{TiO}_2/\text{Al}_2\text{O}_3$  in the final slags established it is necessary only to select the area in the four component system which will yield a low melting zone and in which the aluminum oxide in the final composition would fit into this range of composition. This necessitated considering the compositions that were in the 0, 5, 10 and 20 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  plane. The compositions which fitted in this region are given in Table 2 and Fig 1-4 and are for the region of the highest fluidity and lowest melting points. The data in Table 2 have the greatest bearing on the titaniferous ores which have been investigated.

The weight percent is calculated so that all  $\text{TiO}_2$  in excess is considered to be in solid solution with  $\text{MgO} \cdot 2\text{TiO}_2$ . The areas on the 5 and 10 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  plane are typical for most slags produced from ores. The areas were selected for varying the  $\text{CaO}$  and  $\text{MgO}$  additions because this was the lowest melting and most fluid zone. The slags from the ores would approach the 5 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  with respect to minimum  $\text{TiO}_2/\text{Al}_2\text{O}_3$  and  $\text{TiO}_2/\text{MgO}$ . The flux additions were calculated so that the final slag would have a general composition within these areas, after correcting for  $\text{SiO}_2$  and  $\text{FeO}$  in the slag. The usual practice was to add the minimum amounts of flux. This practice was followed since it was desirable to have as high a  $\text{TiO}_2$  content in the slag as possible.

## Smelting Practice

The investigation was primarily divided into two sections: first, the induction furnace work and second, the single phase arc furnace. Based upon the composition diagram which appeared to give the most fluid slags, a series of mixtures were prepared in which varying amounts of  $\text{CaO}$  and  $\text{MgO}$  were incorporated using petroleum coke as a reducing agent. The use of petroleum coke was practiced in order to avoid any introduction of ash from other types of coke.

The induction furnace utilized for this work was a standard Ajax 6KW high frequency induction furnace,



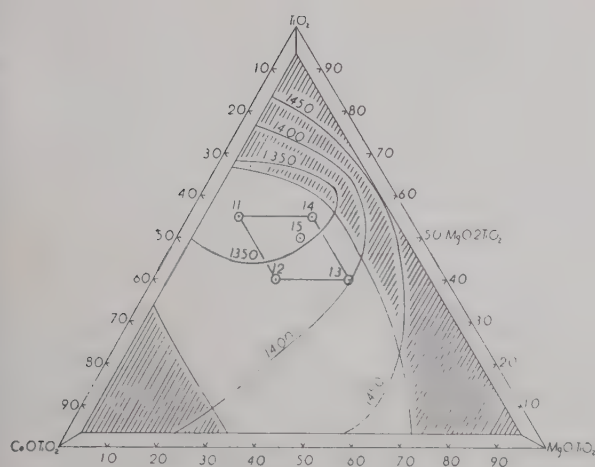


FIG 3—Melting equilibria for system  $\text{CaO} \cdot \text{TiO}_2$ - $\text{MgO} \cdot \text{TiO}_2$ - $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  (10 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  layer).

Clear area—Fluid zone  
Cross hatched area—Viscous zone  
Areas 11, 12, 13, 14—Normal slag zone

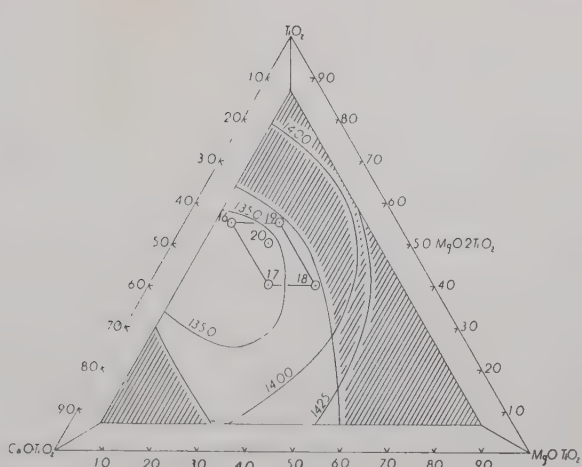


FIG 4—Melting equilibria for system  $\text{CaO} \cdot \text{TiO}_2$ - $\text{MgO} \cdot \text{TiO}_2$ - $\text{TiO}_2$ - $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  (20 mol pct  $\text{Al}_2\text{O}_3 \cdot \text{TiO}_2$  layer).

Clear area—Fluid zone  
Cross hatched area—Viscous zone  
Areas 16, 17, 18, 19—Normal slag zone

using a standard 2 lb graphite crucible (3 in. od  $\times$  2½ in. id  $\times$  3½ in. inside height  $\times$  4 in. outside height). The period of time for a smelting test was varied in order to establish the change in ferrous oxide content of the slag and to observe the change in fluidity as the reduced titanium content increased. Normally the one pound batch required about 30–35 min. to form a molten mixture and this was held in a fluid state for 10–20 min. before removing from the furnace. A holding time of 15 min. was found to be the time required to obtain the necessary reduction of the iron to the metal. The molten slag and metal were poured into a small metal mold and allowed to cool. The metal formed a solid button which was readily separated from the slag above it. The various heats were analysed for TiO<sub>2</sub> and the total ferrous iron as well as carrying out

metallographic, X ray and digestion tests.

## Results Obtained

The results from a series of mixtures which were smelted in order to study the influence of composition upon fluidity of the slag are given in Table 3. The shift in mineral composition is directly related to the amount of trivalent titanium in the slag.<sup>4</sup> The formation of  $\text{Ti(OC)}$  also occurs when the slag is over-reduced. The fluidity of these slags was not noticeably affected by the high  $\text{Ti}^{3+}$  content. Longer holding in the furnace, however, did cause the slags to become viscous and even formed a sintered mass, which was predominantly  $\text{Ti(OC)}$ .

The change in viscosity in the slag is shown in the results given in Table 4 in which the calcium content of the

burden was varied over the range of composition which would give viscous and fluid slags based upon the melting point data observations. The observed fluidity was obtained in accordance with predictions. The calculated slag composition agrees quite well with the observed values.

The maximum and minimum amounts of CaO and MgO required to give suitable slags from various ores was then established. Since the melting point data and fluidity observations appeared to be valid, it was necessary only to delineate the general results for a given ore. These data are given in Table 5 for slags made from the three ores. The maximum amount of MgO that could be used with minimum CaO addition was not established since it was desirable to keep the MgO addition at a minimum. The slags were fluid and furnace behavior was entirely

Table 2 . . . Mineral and Chemical Composition of Low Melting and Fluid Zones

	Mol Pct				Wt Pct			Composition-Wt Pct				TiO <sub>2</sub> /MgO	TiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>
	Al <sub>2</sub> O <sub>3</sub> ·TiO <sub>2</sub>	MgO·TiO <sub>2</sub>	CaO·TiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub> ·TiO <sub>2</sub>	MgO·XTiO <sub>2</sub>	CaO·TiO <sub>2</sub>	TiO <sub>2</sub>	CaO	MgO	Al <sub>2</sub> O <sub>3</sub>		
1	0	5	35	60	0	53	47	78.8	19.2	2.0	0	39.4	
2	0	33	35	32	0	58	42	70.9	17.4	11.7	0	6.1	
3	0	43	15	42	0	81	19	75.8	7.9	16.3	0	4.7	
4	0	25	15	60	0	79	21	81.3	8.5	10.2	0	8.0	
5	0	25	25	50	0	67	33	77.0	13.4	9.6	0	8.0	
6	5	14	29	52	8	45	37	75.0	15.2	5.2	4.8	14.4	15.6
7	5	28	29	38	8	57	35	71.1	14.4	10.0	4.5	7.1	15.8
8	5	38	19	38	8	69	23	72.1	9.6	13.7	4.6	5.2	15.7
9	5	24	19	52	9	66	25	76.1	10.0	9.1	4.8	8.4	15.9
10	5	24	24	47	8	52	30	74.0	12.4	8.9	4.7	8.3	15.7
11	10	9	31.5	49.5	16	46	38	71.8	15.8	3.2	9.2	22.4	7.8
12	10	22.5	31.5	36	16	46	38	68.5	15.1	7.7	8.7	8.9	7.9
13	10	36	18	36	16	63	21	69.7	8.8	12.6	8.9	5.5	7.8
14	10	22.5	18	49.5	17	61	22	73.3	9.2	8.2	9.3	8.9	7.9
15	10	22.5	22.5	45	16	57	27	71.5	11.3	8.1	9.1	8.7	7.9
16	20	8	28	44	30	38	32	67.1	13.1	2.7	17.1	24.9	3.9
17	20	20	28	32	30	39	31	64.6	12.6	6.4	16.4	10.1	3.9
18	20	28	20	32	30	48	22	65.0	9.2	9.2	16.6	7.1	3.9
19	20	16	20	44	31	46	23	67.8	9.5	5.4	17.3	12.6	3.9
20	20	16	24	40	30	43	27	66.6	11.1	5.3	17.0	12.6	3.9



**Table 3 . . . Influence of Fe<sup>2+</sup> and Ti<sup>3+</sup> on Slag Properties**

Burden.....	Baie St. Paul.....					100
	CaO.....					5
	MgO.....					1
	C.....					15

Heat No.	206-4	212-4	208-3	210-3	211-3
TiO <sub>2</sub> Pct.....	58.7	63.2	65.8	67.5	66.4
Fe <sup>2+</sup> Pct.....	9.2	8.3	4.5	2.5	0.7
Ti <sup>3+</sup> Pct.....	0	0.4	3.3	7.8	8.9

Mineral Composition—Pct					
CaO·TiO <sub>2</sub> .....	25	25	30	20	10
(Fe,Mg)O·2TiO <sub>2</sub> .....	45	65	60	40	30
FeO·TiO <sub>2</sub> .....	20	0	0	0	0
Ti(O,C).....	0	0	0	5	5
Glass.....	10	10	10	10	10
MgO·Ti <sub>2</sub> O <sub>3</sub> .....	0	0	0	25	45

**Table 4 . . . Furnace Characteristics of Slag with Variation in CaO Addition**

Burden.....	Baie St. Paul.....					100
	CaO.....					3-7
	MgO.....					1
	C.....					15

CaO.....	3	4	5	6	7
Slag Analysis					
TiO <sub>2</sub> .....	70.5	68.9	67.5	62.0	61.0
Fe <sup>2+</sup> .....	2.5	2.0	2.5	5.0	4.6
Observed Fluidity.....	Cinder	Viscous	V. Fluid	V. Fluid	V. Fluid
Fluidity Expected.....	Sinter	Viscous	Fluid	Fluid	Fluid

Mineral Composition—Pct					
CaO·TiO <sub>2</sub> .....	10	20	30	40	45
(Fe,Mg)O·2TiO <sub>2</sub> .....	80	70	60	50	45
Glass.....	10	10	10	10	10

Calculated Chemical Composition—Pct					
TiO <sub>2</sub> .....	70.3	69.0	67.8	65.0	63.9
Fe <sup>2+</sup> .....	2.5	2.5	2.4	4.9	4.8
CaO.....	7.1	8.7	10.3	11.6	12.7
MgO.....	8.7	8.5	8.4	8.0	7.9
Al <sub>2</sub> O <sub>3</sub> .....	5.1	5.0	4.9	4.7	4.6

satisfactory. This series indicated that within limits a slag could be prepared of desired composition with proper fluidity if within the area of fluidity as indicated by the melting point diagrams. The absolute maximum amount of fluxes was not determined since the objective was to produce a high titanium slag.

## Arc Furnace Smelting

The induction furnace tests had shown that the formation of a fluid slag which had the necessary chemical, mineralogical and digestible characteristics could be accomplished. The application of the observations of the induction furnace to arc furnace practice was the next step. A small arc furnace was utilized which was a modi-

fied Pittsburgh Lectromelt size "X" single phase unit. The power supply was a 440 volt primary transformer with variable secondary taps. The taps used for this smelting work was 78 volts with 60 pct reactance. The usual operation of the furnace was about 40 KVA utilizing a 1¼ in. electrode. The input to the furnace was controlled by manually raising and lowering the electrode.

The furnace was lined with graphite blocks forming a hearth approximately 80 sq in. and had a depth of about 9 in. The furnace was covered and an opening for charging the furnace was allowed through the top. The slag and metal were removed from the furnace through a tap hole in the bottom.

Since the ores of primary interest were the Canadian ore from Baie St. Paul and the MacIntyre ilmenite con-

**Table 5 . . . Fluid Slags from Various Ores—Maximum and Minimum Flux Addition**

Ore	MacIntyre		Baie St. Paul	
Burden				
Ore.....	100	100	100	100
CaO.....	6	8	4	7
MgO.....	1	5	0	3
C.....	15	15	15	15
Slag Analysis				
TiO <sub>2</sub> .....	70.8	63.5	67.4	59.2
Fe <sup>2+</sup> .....	4.9	4.0	4.2	5.5

Mineral Composition—Pct				
CaO·TiO <sub>2</sub> .....	20	50	25	40
(Fe,Mg)O·2-TiO <sub>2</sub> ..	70	40	65	50
Glass.....	10	10	10	10

Calculated Chemical Composition—Pct				
TiO <sub>2</sub> .....	68.6	62.8	69.0	61.8
Fe <sup>2+</sup> .....	4.6	3.9	4.3	4.6
CaO.....	10.2	12.4	8.7	12.3
MgO.....	5.5	10.7	6.8	10.8
Al <sub>2</sub> O <sub>3</sub> .....	4.8	4.5	5.0	4.5
SiO <sub>2</sub> .....	6.1	5.6	6.2	5.6



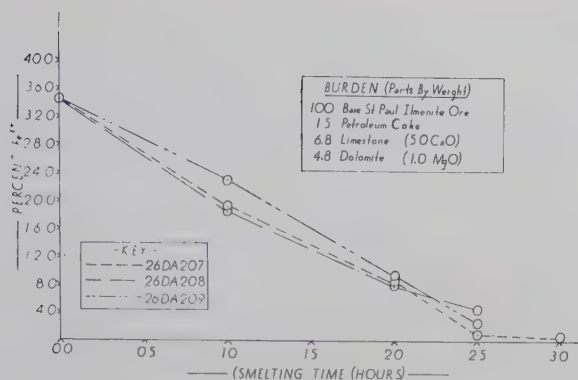


FIG 5—Progressive reduction of Baie St. Paul ore during smelting in arc furnace.

centrate the arc smelting was limited to these two materials. The burdens selected for the tests were those which had shown the best properties from induction furnace tests and were as follows:

	Baie St. Paul, Pounds	MacIntyre, Pounds
Ore.....	50	50
Petroleum coke.....	7.5	7.5
Limestone.....	3.4	4.3
Dolomite.....	2.4	2.4

The flux addition is equivalent to 5.0 parts of CaO and 1 part of MgO added to the burden for Baie St. Paul and 6.0 parts CaO and 1 part MgO for MacIntyre.

It was a regular practice to heat the furnace first by arcing the electrodes on a carbon block for about  $\frac{1}{2}$  hr prior to adding the charge. Approximately  $\frac{1}{3}$  of the charge was dumped into the furnace after removing the carbon block and melted down. The balance of the charge was added gradually as the melting proceeded. The total time required for charging varied from 20–30 min. In order to establish the time required to obtain the necessary reduction, samples of slags were taken at various intervals for analyses. The total smelting time varied from 2–3 hr, depending upon the general operating procedure. The temperature of the slag as it flowed from the furnace, observed with an optical pyrometer, was normally about 1500°C. The slag was tapped into a ladle in which iron settled out in the bottom and the slag formed as a separate layer on top. The slags

which had proven most satisfactory in the induction furnace could be duplicated on a larger scale in the arc furnace.

The progressive reduction of the charge in several tests made from Baie St. Paul ilmenite is given in Fig 5. The reproducibility of the smelting was fairly consistent when the power input was maintained very carefully.

Observation of the mechanics of the reduction definitely indicated that the slag and carbon were reacting. The reduction was found to be affected by type of carbon used. Coke, because of its high surface area, was better suited for a reductant than anthracite. As a result of the high secondary voltage delivered by the transformer, the electrodes could not be maintained in the slag during the final period of reduction. The change in resistivity of the slag was quite apparent and appeared to be a function of the ferrous iron content.

### Summary

1. The adaptation of melting point data to slag formation is shown.
2. Ilmenites having 38–44 pct  $\text{TiO}_2$  were smelted in laboratory to produce fluid slags with 65–69 pct  $\text{TiO}_2$ .
3. A high titanium slag can be produced in which the formation of appreciable amounts of  $\text{Ti}^{3+}$  may be avoided.
4. The final slag with 62–69 pct  $\text{TiO}_2$  requires 5–11 pct MgO and 9–13 pct CaO in order to have desired fluidity from MacIntyre and Baie St.

Paul ilmenites.

5. High titanium slags can be produced in an arc furnace with slag temperatures of 1450–1550°C which were identical in behavior and properties to the induction furnace slags.

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# Petrology of High Titanium Slags

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## Introduction

Extensive studies have been carried out on electric furnace and blast furnace slags obtained in the winning of iron from its ores. These slags normally consist of elements of the gangue minerals present in the ores, as well as the added flux materials. In consequence, melts of  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  can be considered as representing typical slag compositions. When a slag of this composition cools, it usually crystallizes according to predictions possible from an equilibrium diagram of these constituents, providing the melt is not undercooled to form glass. The melt is either viscous or fluid, depending upon the ratio of binary cations to silica, and crystallizes easily or forms a glass for the same reasons. If the melt is not overheated so that carbides of the metal components of the slag are formed and if the composition of the slag is so adjusted that it has a high fluidity, liquid equilibrium is attained and the slag can be held in a liquid state for extended periods of time. Upon tapping, the slag crystallizes into minerals, the type and proportion of which are determined by the melt composition. Since equilibrium is attained, the hold-period is not critical.

In melts containing a large increment of titanium, however, the normal slag procedures are not applicable. Titanium, as one of the atomic transition elements, is, at elevated temperatures, capable of being reduced to form metalloid compounds much more readily than the refractory oxides present in normal slags. In consequence, an oxide melt containing titanium never reaches equilibrium in a reducing environment, but continues to shift its composition until cooled. If melts of this nature are cooled and samples submitted to metallographic and X ray analysis the course

of reaction and crystallization in this type of slag can be determined.

## Preparation of Slag

The slags investigated fell into the system  $\text{CaO-MgO-TiO}_2\text{-Al}_2\text{O}_3\text{-SiO}_2$  and were produced from ilmenite ores reduced by carbon in an electric furnace. Since the equilibrium series<sup>1</sup> and the laboratory smelting of ilmenite<sup>2</sup> are described in two of the accompanying papers, detailed description of the smelting procedure is not required here. However, certain essentials must be mentioned.

Two types of melts were used to produce slags studied in this investigation. The first series of smelts made to determine proper flux addition were produced in a 4 lb Ajax induction furnace. The charge, consisting of ore with the proper flux addition, was heated in a graphite crucible until fluid, held fluid for a sufficient time period to obtain 1-5 pct  $\text{FeO}$  content, and poured. Because of the small size of the charge only the final sample of these melts could be examined. In the melts made in the 50 lb arc furnace, however, grab samples taken at 10 min. intervals between time of initial melting and final pouring were available for examination. These samples allowed a much clearer picture of the course of reaction and crystallization.

## Sample Preparation

Any material containing appreciable

amounts of ferrous oxide and reduced titanium compounds is opaque to transmitted light. Therefore, all petrographic studies had to be made on polished slag sections. A representative sample of slag was cut or broken, mounted in a thermosetting plastic, ground flat using 400 grit silicon carbide, the coarse scratches removed with 600 grit silicon carbide and polished on billiard cloth using levigated alumina. Rouge was avoided because of the entrainment of the red particles in pores in the slag, causing a possible confusion with some of the mineral phases. In order to prevent sample projection above the plastic surface red bakelite was used to hold the sample, and backed up with clear lucite. In this manner sample labels could be permanently retained in the mounting. The polished samples were examined on a Bausch and Lomb metallograph at magnifications of 250 $\times$ , 500 $\times$ , 1000 $\times$  and 1800 $\times$ . The instrument was equipped for examination of specimens under bright field illumination and with crossed nicols. A magenta tint plate to aid in color tone differentiation was also used.

## Petrology of Slags

In order to determine the composition and mineral relations of a previously unreported system petrologically, it is essential that the starting composition, reaction temperature and final composition be known. The chemical composition of the ilmenite ore used in these smelts is given in Table 1, and the complete analysis of a typical high titanium, low iron slag is given in Table 2.

In the winning of  $\text{TiO}_2$  from ilmenite by a smelting process it is necessary to produce a slag which will melt at an economically feasible temperature, remain molten as the iron is removed by reduction, be fluid enough to be readily removed from the furnace, contain a high percentage of  $\text{TiO}_2$  and a low percentage of reduced titanium com-

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<sup>1</sup> References are at the end of the paper.



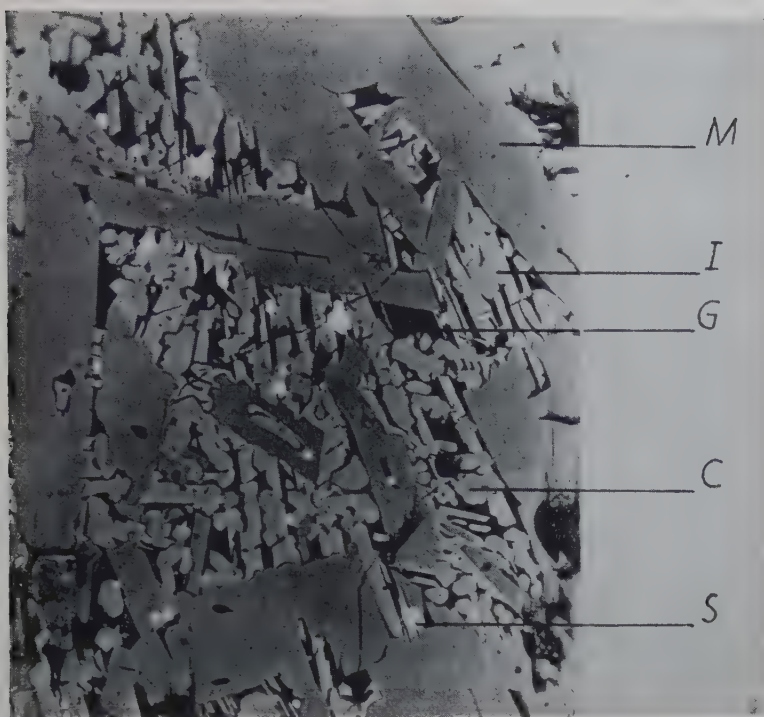


FIG 1—Micrograph of slag sample No. 1.  $\times 500$ .

Slightly reduced in reproduction. The slag at this early stage of reduction contains 16.2 pct FeO and 58.8 pct  $\text{TiO}_2$ . The presence of this amount of ilmenite indicates that reduction of the slag has not progressed to the desired final composition. Mineralogical composition is as follows:

I—20 pct ilmenite  
C—10–20 pct  $\text{CaO} \cdot \text{TiO}_2$   
M—50–60 pct  $(\text{Fe}, \text{Mg})\text{O} \cdot 2 \text{TiO}_2$   
G—10 pct silicate glass  
S—Small amount Fe, FeS, etc.

1000°C, by a solid state reaction, leaving a composition that appears to be  $\text{FeO} \cdot 2\text{TiO}_2$ . At 1150°C, and when the amount of  $\text{FeO} \cdot 2\text{TiO}_2$  reaches 70 pct an apparent eutectic occurs in the system  $\text{FeO} \cdot \text{TiO}_2$ – $\text{FeO} \cdot 2\text{TiO}_2$  and the charge becomes pasty.\*

If reduction continues, the charge remains pasty until the temperature reaches 1450°C. Here a probable equilibrium between  $\text{FeO} \cdot \text{TiO}_2$ ,  $(\text{Fe}, \text{Mg})\text{O} \cdot 2\text{TiO}_2$  and calcium magnesium silicate makes the melt fluid. If frozen at this point, the slag consists of ilmenite,  $\text{FeO} \cdot 2\text{TiO}_2$  (probably with some magnesium replacing the iron), minor amounts of  $\text{CaTiO}_3$  and a glass. The FeO content of such a slag is 11–14 pct. If reduced further the last skeletal crystals of ilmenite disappear at 7–8 pct FeO and the slag consists of a  $(\text{Fe}, \text{Mg})\text{O} \cdot 2\text{TiO}_2$  structure and  $\text{CaTiO}_3$  (near the 60–40 eutectic proportion)

\*Grieve and White<sup>3</sup> indicate two eutectics and only pseudobrookite and ilmenite in the thermal equilibrium diagram  $\text{FeO} \cdot \text{TiO}_2$ . However, they mention an undetermined amount of  $\text{Fe}_2\text{O}_3$  in their system. We have found that if conditions are sufficiently oxidizing to transform FeO to  $\text{Fe}_2\text{O}_3$ , the compound  $\text{FeO} \cdot 2\text{TiO}_2$  does not form, being replaced by ilmenite or pseudobrookite and rutile. If the compound  $\text{FeO} \cdot 2\text{TiO}_2$  (with minor amounts of magnesium in solid solution) is heated to 500°C in air it dissociates to pseudobrookite and *analase*. This indicates that the  $(\text{Fe}, \text{Mg})\text{O} \cdot 2\text{TiO}_2$  structure is stable only when deficient in oxygen.

Table 1 . . . Chemical Composition of Baie St. Paul Ilmenite (Weight, Pct)

$\text{TiO}_2$ .....	39.7
FeO.....	28.6
$\text{Fe}_2\text{O}_3$ .....	20.6
CaO.....	0.7
MgO.....	3.6
$\text{Al}_2\text{O}_3$ .....	2.4
$\text{SiO}_2$ .....	3.4
$\text{V}_2\text{O}_5$ .....	0.32
$\text{Cr}_2\text{O}_3$ .....	0.11
$\text{P}_2\text{O}_5$ .....	0.045

Table 2 . . . Chemical Composition of Typical High  $\text{TiO}_2$ —Low FeO Slag (Weight, Pct)

$\text{TiO}_2$ .....	68.5
$\text{Ti}^{+3}$ .....	5.1
FeO.....	3.1
CaO.....	2.5
MgO.....	7.2
$\text{Al}_2\text{O}_3$ .....	5.4
$\text{SiO}_2$ .....	5.1
S.....	0.91
$\text{V}_2\text{O}_5$ .....	0.32
C.....	0.06

pounds, and be amenable to digestion in sulphuric acid. In light of the melting data obtained in the accompanying paper on slag equilibria it is apparent that at least part of these requirements was fulfilled by the additions of lime and magnesia fluxes to ilmenite.

## Course of Reduction

As ilmenite is heated with carbon, the iron begins to reduce to metal at

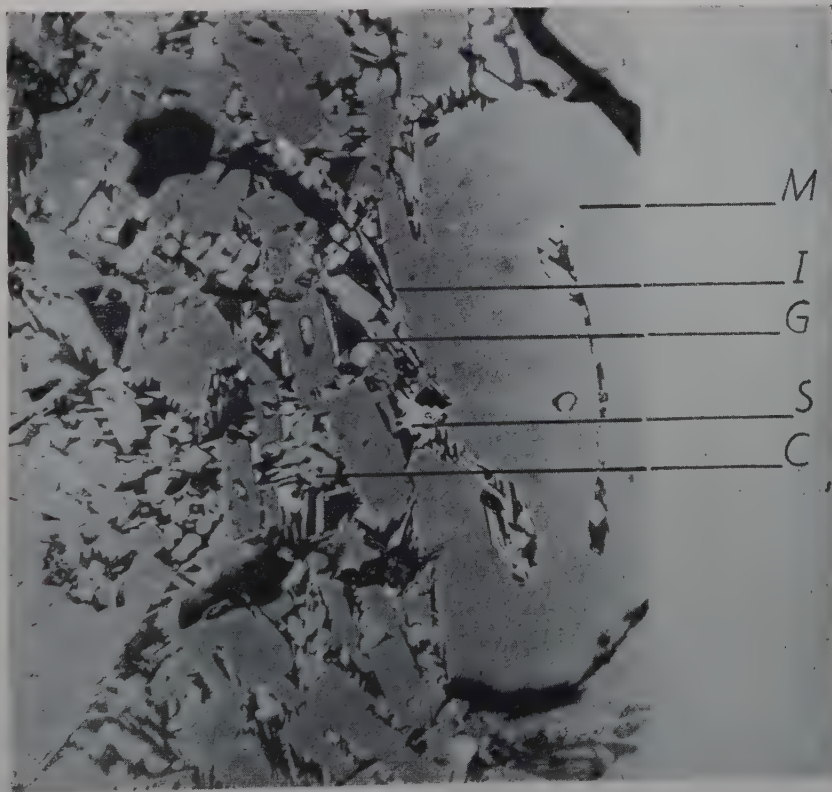


FIG 2—Micrograph of slag sample No. 2.  $\times 500$ .

Slightly reduced in reproduction. The slag at this stage of reduction contains 13.3 pct FeO and 60.3 pct  $\text{TiO}_2$ . Ilmenite still remains in sufficient quantity as acicular particles to indicate the reduction has not progressed to the optimum composition. The mineralogical composition is as follows:

I—10–15 pct ilmenite  
C—10–20 pct  $\text{CaO} \cdot \text{TiO}_2$   
M—50–60 pct  $(\text{Fe}, \text{Mg})\text{O} \cdot 2\text{TiO}_2$   
G—10 pct silicate glass  
S—Small amount of Fe, FeS, etc.



and glass.  $(\text{Fe,Mg})\text{O} \cdot 2\text{TiO}_2$  is the primary phase,  $\text{CaTiO}_3$  the secondary phase and finally, an interstitial glass, which may on slow cooling partially devitrify to poorly crystallized silicates. As reduction proceeds, the mineralogical relationship of the constituents remains essentially the same except that iron is isomorphously replaced by magnesium in the compound  $(\text{Fe,Mg})\text{O} \cdot 2\text{TiO}_2$ . However, as the iron content of the slag system falls below 3 pct the titanium begins to reduce. If frozen here the slag is still amenable to processing, but the compound is designated as  $\text{MgTi}_3\text{O}_6$ . As the loss of oxygen from the system continues the  $\text{MgO} \cdot 2\text{TiO}_2$  type structure is no longer stable and shifts to a structure designated as  $\text{MgTi}_2\text{O}_4$ . When this occurs, the melt becomes more refractory and hence less fluid and is no longer amenable to standard sulphuric acid digestion procedure. The slag is then called "over-reduced." If reduction is allowed to continue  $\text{Ti}(\text{O,C})$  and finally  $\text{TiC}$ , are formed and the melt freezes. As far as can be determined, the amount and composition of the  $\text{CaTiO}_3$  remains constant, from its first crystallization in eutectic proportions, throughout the course of reduc-

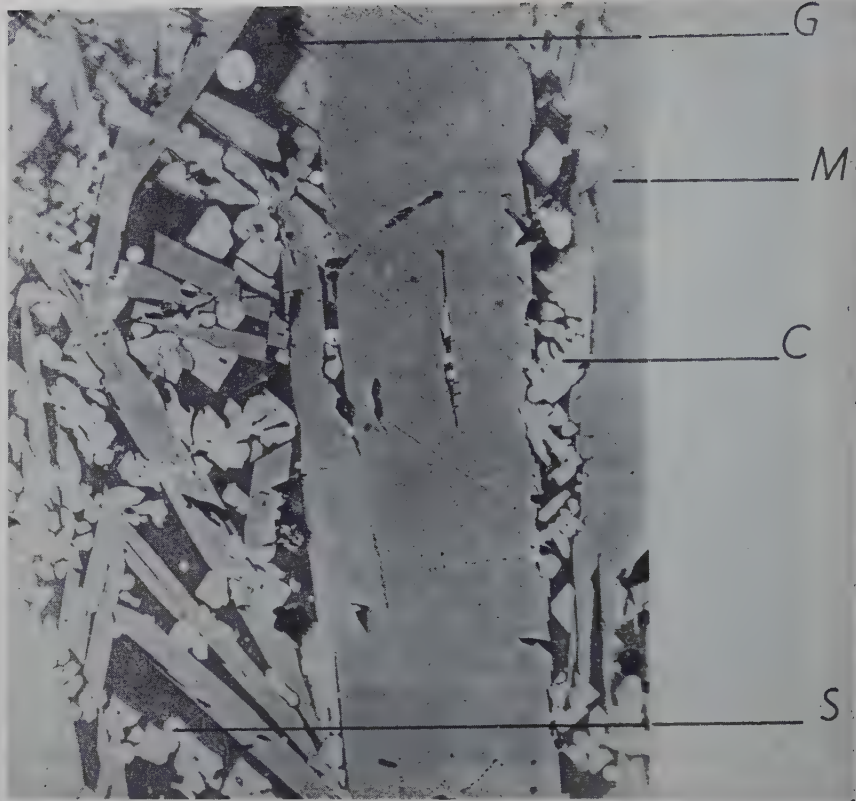


FIG 4—Micrograph of slag sample No. 4.  $\times 500$ .

At this stage of reduction the slag contains 4.9 pct FeO and 67.8 pct  $\text{TiO}_2$ . The presence of the  $\text{MgTi}_3\text{O}_6$  phase indicates slight over-reduction of the  $(\text{Fe,Mg})\text{O} \cdot 2\text{TiO}_2$  phase which apparently is not detrimental to optimum digestion of the slag. Mineralogical composition is as follows:

C—20–30 pct  $\text{CaO} \cdot \text{TiO}_2$   
M—60 pct  $(\text{Fe,Mg})\text{O} \cdot 2\text{TiO}_2$   
5 pct  $\text{MgTi}_3\text{O}_6$  (not evident at  $\times 500$ )  
G—10 pct silicate glass  
S—Small amount Fe, FeS, etc.

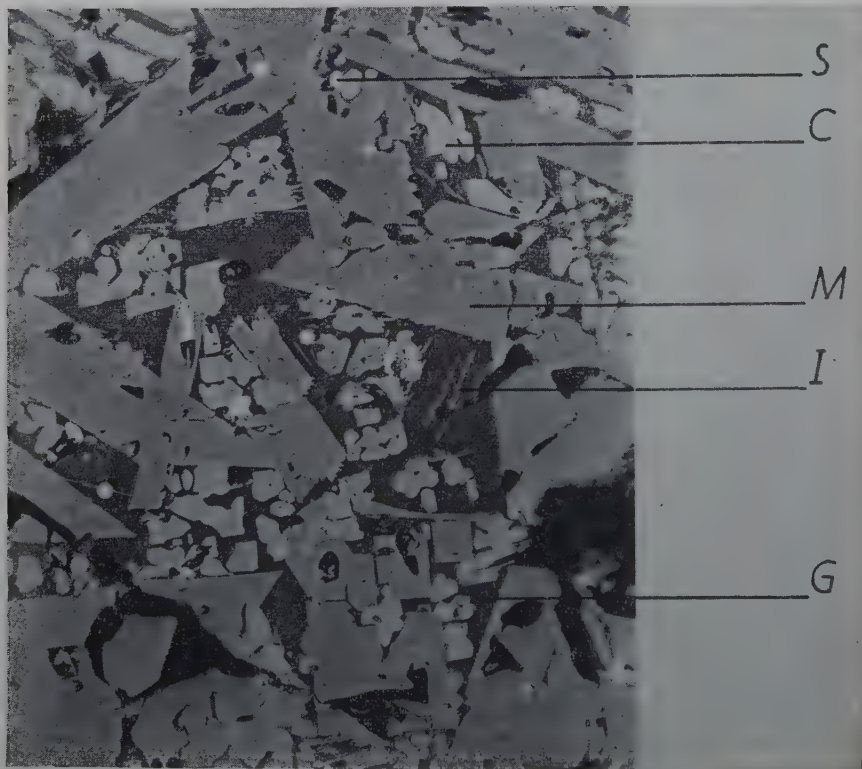


FIG 3—Micrograph of slag sample No. 3.  $\times 500$ .

The slag now contains 8.7 pct FeO and 63.8 pct  $\text{TiO}_2$ . It has now been reduced almost to the optimum composition since ilmenite remains only as small crystals surrounding the  $(\text{Fe,Mg})\text{O} \cdot 2\text{TiO}_2$  phase and no over-reduction of the dititanate phase is evident. The mineralogical composition is as follows:

I—5 pct ilmenite  
C—20–30 pct  $\text{CaO} \cdot \text{TiO}_2$   
M—50–60 pct  $(\text{Fe,Mg})\text{O} \cdot 2\text{TiO}_2$   
G—10 pct silicate glass  
S—Small amount Fe, FeS, etc.

tion. The series of micrographs shown in Fig 1 through 5 illustrates the course of reduction but does not in all cases indicate the proportions of phases present in the melts. The representative phase proportions were determined from a large number of samples.

### Mineral Phases

The isomorphous series  $\text{FeO} \cdot 2\text{TiO}_2$ – $\text{MgO} \cdot 2\text{TiO}_2$  is by far the most interesting mineral phase in this type of slag. It not only is the primary crystallization phase in the slags, but incorporates the greatest amount of titanium in the system. The shift from  $\text{FeO} \cdot 2\text{TiO}_2$  to  $(\text{Fe,Mg})\text{O} \cdot 2\text{TiO}_2$  is continuous and occurs as iron is removed from the system. This composition shift is difficult to detect. It is distinguished microscopically only by a change in color of the laths from dark gray to light gray as the amount of iron is decreased. X ray powder patterns of  $\text{FeO} \cdot 2\text{TiO}_2$  and  $(\text{Fe,Mg})\text{O} \cdot 2\text{TiO}_2$  are apparently identical.

Under reducing conditions  $\text{MgO} \cdot 2\text{TiO}_2$  is not stable and always contains vacant titanium equipoints. As



mentioned above, the formula  $MgTi_3O_6$  is assumed and is used to indicate this. The structure and habits of this compound are identical with  $MgO \cdot 2TiO_2$  and are not distinguishable by X ray powder patterns. Under reflected light the color is a very light gray with a pink tone.

The next reduction stage, however, is readily distinguished under the microscope. It is a dark red to purple in reflected light and assumes a quadratic habit. Although the structure has not been completely delineated it is apparently cubic and has been assigned the probable formula  $MgTi_2O_4$ .

The flexibility of the structure which is called " $MgO \cdot 2TiO_2$  type" is indicated by its ability to lose iron, gain magnesium and lose oxygen, and still preserve its identity.

There are indications that it is capable of far more flexibility. It was shown early in the investigation that the amount of  $TiO_2$  determined chemically to exist in these slags was not accounted for by the  $(Fe,Mg)O \cdot 2TiO_2$  and  $CaTiO_3$  present. In consequence a series of synthetic slags was prepared under both reducing and oxidizing conditions in which the proportions  $MgO$  to  $TiO_2$  were varied.

Under oxidizing conditions, no success has been obtained in attempts to form magnesium titanates higher in  $TiO_2$  than  $MgO \cdot 2TiO_2$ . All attempts to form  $MgO \cdot 3TiO_2$  have given  $MgO \cdot 2TiO_2$  and rutile.

Under strongly reducing conditions, however, it has been found that the  $(Fe,Mg)O \cdot 2TiO_2$  structure will take  $TiO_2$  into solid solution up to explored limits of the order of 12 mol of  $TiO_2$  per mol of  $(Fe,Mg)O$ , without a breakdown in structure. The solid solution compound with  $TiO_2$  values of this order has been produced readily during smelting of low gangue ilmenite ores and also from mixtures of  $Fe_2O_3$ ,  $MgO$  and  $TiO_2$ . X ray examination of the compound shows a shift in the structure to larger interplanar spacings (for example,  $(Fe,Mg)O \cdot 12TiO_2$  showed a strong line at  $1.894\text{\AA}$  in contrast to the typical line at  $1.876\text{\AA}$  in  $MgO \cdot 2TiO_2$  and  $(Fe,Mg)O \cdot 2TiO_2$ ).

Oxidation of the  $(Fe,Mg)O \cdot 2TiO_2$ - $(Fe,Mg)O \cdot 12TiO_2$  series in air at  $500^\circ\text{C}$  for several hours causes a breakdown in the structure to the compounds which are stable under oxidizing conditions. These compounds are  $Fe_2O_3$ ,  $TiO_2$ ,  $MgO \cdot 2TiO_2$ ,  $MgO \cdot TiO_2$  and  $TiO_2$ . At  $500\text{--}800^\circ\text{C}$  the  $TiO_2$  is present as anatase. At temperatures higher than  $800^\circ\text{C}$ , the  $TiO_2$  which is crys-

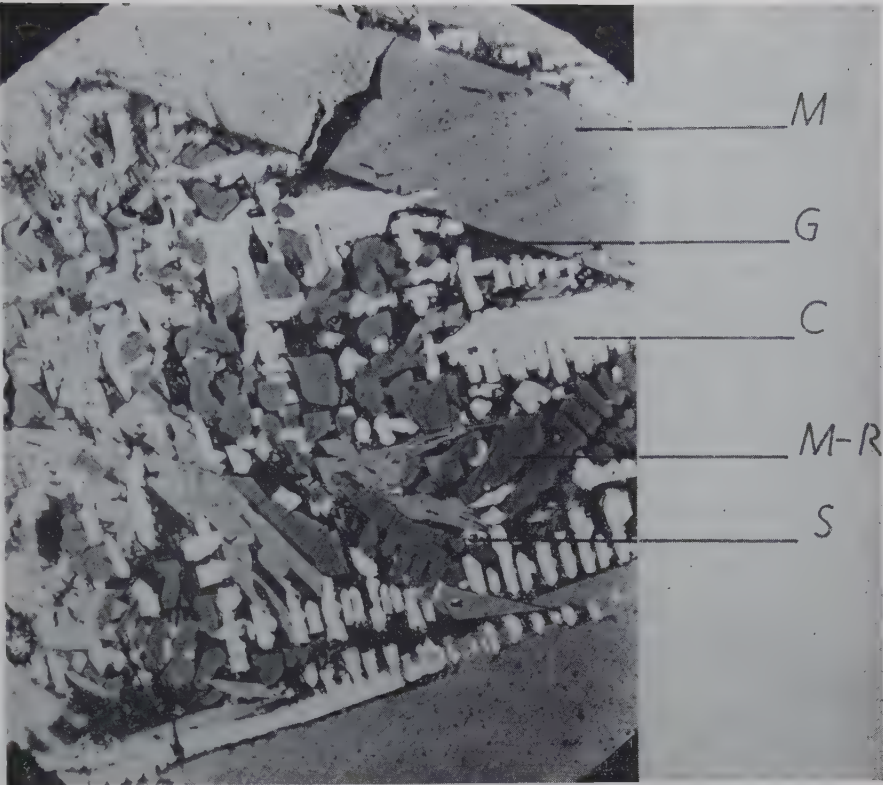


FIG 5—Micrograph of slag sample No. 5.  $\times 500$ .  
At this stage of reduction the slag contains 1.0 pct FeO and 70.7 pct  $TiO_2$ . The presence of the  $MgTi_2O_4$  stage indicates serious over-reduction of the  $(Fe,Mg)O \cdot 2TiO_2$  phase and this phase is detrimental to optimum digestion. Mineralogical composition is as follows:  
C—20 pct  $CaO \cdot TiO_2$   
M—20–30 pct  $(Fe,Mg)O \cdot 2TiO_2$   
M-R—30–40 pct  $MgTi_2O_4$   
G—15 pct silicate glass  
S—Small amount Fe, FeS, etc.

tallized from the  $(Fe,Mg)O \cdot 2TiO_2$  structure exists as rutile.

The fact that  $TiO_2$  cannot be retained in the structure under oxidizing conditions indicates that the solid solution of  $TiO_2$  in the dititanate structure is possible under reducing conditions because of an oxygen deficient lattice in the dititanate. Data on the degree of oxygen deficiencies in the lattice have not been completely determined.

Table 3 . . . Patterns of Titanates. Spectrometer, Cu,  $K\alpha$ ,  $\lambda = 1.5374\text{\AA}$ , 2 Rpm Scan. No Corrections Applied

$MgO \cdot 2TiO_2$ or $(Fe,Mg)O \cdot 2TiO_2$		50 Mol Pct $MgO \cdot 2TiO_2$ 50 Mol Pct $Al_2O_3 \cdot TiO_2$ (oxidizing conditions)		$Al_2O_3 \cdot TiO_2$ (oxidizing conditions)		$(Fe,Mg)O \cdot 2TiO_2$ + 20 Pct $Al_2O_3$ (by wt.) with $TiO_2$ in Solid Soln. (reducing conditions)		$(Fe,Mg)O \cdot 2TiO_2$ structure (Empirical Formula $(Fe,Mg)O \cdot 12TiO_2$ ) (reducing conditions)	
$d/n, \text{\AA}$	$I/I_1$	$d/n, \text{\AA}$	$I/I_1$	$d/n, \text{\AA}$	$I/I_1$	$d/n, \text{\AA}$	$I/I_1$	$d/n, \text{\AA}$	$I/I_1$
5.0	0.12	4.83	0.38w	4.71	0.47	4.86	0.4	4.97	0.23w
4.9	0.12								
3.51	1.00	3.43	1.00	3.35	1.00	3.44	1.00	3.54	1.00
2.86	0.09								
2.75	0.6	2.71	0.8	2.65	0.7	2.71	0.9	2.76	0.6
2.45	0.2					2.43	0.2	2.47	0.23
2.22	0.15	2.40	0.20w	2.36	0.20	2.37	0.15	2.43	0.19
2.19	0.2	2.18	0.22	2.14	0.31	2.20	0.2	2.24	0.22
1.967	0.3	2.16	0.20	2.11	0.30	2.17	0.2	2.21	0.15
1.876	0.6	1.939	0.34w	1.897	0.52	1.951	0.24	1.98	0.25
1.846	0.15	1.829	0.43w	1.792	0.44	1.854	0.35	1.894	0.75
1.754	0.15					1.839	0.1	1.857	0.15
1.665	0.15	1.723	0.14w	1.687	0.28	1.73	0.1w	1.763	0.15
1.634	0.2	1.645	0.11	1.606	0.27	1.648	0.1	1.67	0.10w
		1.632	0.11	1.578	0.28	1.621	0.2	1.65	0.15w
		1.606	0.16	1.55	0.08w	1.598	0.1		
1.551	0.3			1.519	0.06			1.559	0.32
1.535	0.2	1.515	0.29w	1.484	0.36	1.530	0.3	1.542	0.10
1.424	0.09	1.400	0.09w	1.449	0.08	1.413	0.1	1.43	0.10w
1.378	0.08	1.352	0.11w	1.371	0.14	1.364	0.1	1.37	0.10w
1.358	0.15			1.32	0.09w	1.350	0.2		
1.318	0.08			1.305	0.16				
1.265	0.10			1.266	0.11	1.251	0.1	1.268	0.08
1.245	0.10			1.253	0.11			1.251	0.07
				1.219	0.09				

w — after relative intensity means wide line or lines.



No Corrections Applied

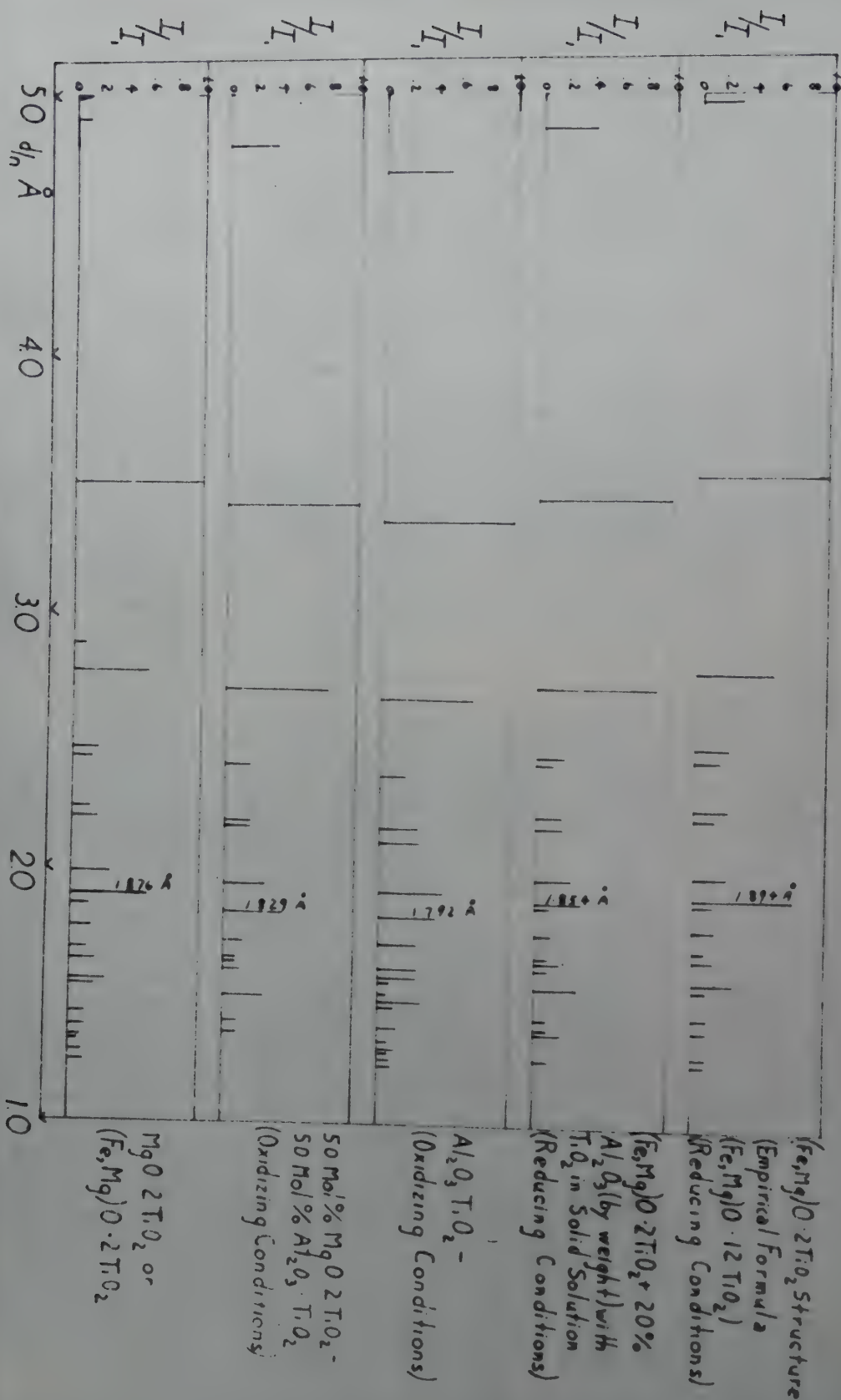


FIG 6—Patterns of Titanates.



Table 4 . . . Properties in Reflected Light of Phases Present in High Titanium Slags Made by Fluxing Ilmenite with CaO and MgO

Phase	Bright Light with Blue Filter Screen		Crossed Nicols		Sensitive Tint Plate	
	Color	Habit	Color	Habit	Color	Habit
CaO·TiO <sub>2</sub> .....	Light gray to white	Dendritic aggregates or individual blocky crystals.	Very bright white to brown	Birefringent in all positions of stage.	Highly reflecting blue	Blue in all positions of stage.
FeO·TiO <sub>2</sub> ..... (Ilmenite)	Light gray to pale brown	Occurs as laths in glass or as rims around (Fe,Mg)O·2TiO <sub>2</sub>	Black to dark gray	Extinct parallel to horizontal cross hair. Gray parallel to vertical cross hair.	Pink to blue	Pink parallel to horizontal cross hair. Blue parallel to vertical cross hair.
(Fe,Mg)O·2TiO <sub>2</sub> .....	Light to dark gray	Well defined laths	Dark gray to black	Extinct parallel to vertical cross hair. Gray parallel to horizontal cross hair.	Pink to blue	Pink parallel to vertical cross hair. Blue parallel to horizontal cross hair.
MgTi <sub>2</sub> O <sub>6</sub> .....	Gray black to light brown	Well defined laths	Black to light brown	Extinct parallel to vertical cross hair. Light brown parallel to horizontal cross hair.	Pink to blue	Pink parallel to vertical cross hair. Blue parallel to horizontal cross hair.
MgTi <sub>2</sub> O <sub>4</sub> .....	Brownish red	Quadratic crystals	Black to brown	Extinct in 90° positions. Brown in 45° positions.		
Glass.....	Dark gray	Fills interstices of crystals.	Black	Black in all positions of stage.		

Under oxidizing conditions there is a continuous solid solution series Al<sub>2</sub>O<sub>3</sub>·TiO<sub>2</sub>-MgO·2TiO<sub>2</sub> as discussed in the accompanying melting equilibria paper. Under reducing conditions Al<sub>2</sub>O<sub>3</sub> is taken into solid solution in the (Fe,Mg)O·2TiO<sub>2</sub> structure up to approximately 20 pct by weight, without the formation of Al<sub>2</sub>O<sub>3</sub>·TiO<sub>2</sub>. This solid solution compound shows a considerable shift in the structure to smaller interplanar spacing (for example, the shift is from 1.876Å for normal MgO·2TiO<sub>2</sub> or (Fe,Mg)O·2TiO<sub>2</sub> to 1.854Å for (Fe,Mg)O·2TiO<sub>2</sub> with 20 pct Al<sub>2</sub>O<sub>3</sub> (by weight) present in solid solution. Fig 6 shows X ray powder pattern comparisons of the effects of solid solution of TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> on the (Fe,Mg)O·2TiO<sub>2</sub> structure. Additions of over 20 pct Al<sub>2</sub>O<sub>3</sub> gave the above solid solution compound and Al<sub>2</sub>O<sub>3</sub>·TiO<sub>2</sub>. The line position for Al<sub>2</sub>O<sub>3</sub>·TiO<sub>2</sub> in this region of the X ray pattern is 1.792Å. Table 3 shows the *d/n* values for these compounds.

No change in the structure of the compound from the normal (Fe,Mg)O·2TiO<sub>2</sub> is discernible under the metallographic microscope when TiO<sub>2</sub> or

Al<sub>2</sub>O<sub>3</sub> are present in solid solution even at the higher values of these oxides.

CaTiO<sub>3</sub> apparently always crystallizes as the secondary phase in the range of composition used in slag formation. These crystals are cubic in crystallization, quadratic in habit and usually the only colorless constituent of the slag. In slag samples which were quickly frozen the CaTiO<sub>3</sub> occurs as skeletal outlines in the glass. In slowly cooled slags the large cubic crystals are fully developed. No evidence of structural shift or solid solution in CaTiO<sub>3</sub> was observed at any reduction stage by either X ray diffraction patterns or petrographic character.

The role of the silicate glass in these melts is rather ambiguous. It apparently helps fluidization of the melt by incorporating at least part of the alumina to form a probable FeO-CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> melt. The index of refraction of the glass preserved in the quickly frozen slags is ±1.55. The silicates which are observed in the slowly cooled slags are so poorly crystallized that their identity could not be determined.

Table 4 is a compilation of the opti-

cal properties in reflected light of the phases which occur in titanium bearing slags when fluxed with lime and magnesia.

### Acknowledgment

The writers wish to express thanks to Dr. S. S. Cole, Assistant Research Manager, Titanium Division, National Lead Co., for technical suggestions and aid in preparation of the paper and to Mr. C. H. North of the Research Laboratory for the X ray data which have been of great value in this investigation.

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## Notes on the Electrolytic Isolation of Carbides in Steel

GUSTAF WRANGLÉN\*

On account of the possibility of isolating carbides in steel it is generally assumed that the electrode potential of iron carbide is more noble than that of ferrite.<sup>1,2,3,4,5</sup> Differences from 0.032 V<sup>1</sup> to 0.4 V<sup>4</sup> have been recorded. However, it is important to realize that the more noble behavior of

cementite in relation to ferrite is not

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<sup>1</sup> References are at the end of the paper.

an issue of thermodynamics but of reaction kinetics. Theoretically, cementite is less noble than ferrite, which is a direct consequence of its thermodynamic instability. With the value ΔF<sub>298</sub><sup>o</sup> = +4700 cal per mol for the free energy of formation of cementite<sup>6</sup> it is easily calculated that the electrode



potential of the reaction  $\text{Fe}_3\text{C} = 3\text{Fe}^{2+} + \text{C} + 6e^-$ , found to be the relevant one, particularly in neutral solution,<sup>5</sup> is  $-0.47$  V (cf.  $e^\circ_{\text{Fe}} = 0.44$  V). There are great hindrances to this reaction, however, implying a slow reactivity, which has to be termed high chemical polarization in this case. Hence, the electrolytic isolation of iron carbide is *not* a parallel to the retention of noble metals as an anode slime in electrolytic refining.

It is evident, furthermore, that the reaction referred to cannot be even chemically reversible, the less so thermodynamically. An equilibrium corresponding to the computed figure will never be reached. On measurement more noble and varying values will be obtained. These static potentials are of minor interest. It would be more valuable to determine the actual dissolution potentials during electrolysis and then select those conditions for separation, under which the difference between the dissolution potentials of cementite and ferrite is the greatest.

Structure is the most important factor influencing the isolation. If the carbide is present as large spheroid particles it is fairly insensible to varying conditions of electrolysis. If the structure is unfavorable (lamellar pearlite, cementite network) the operating conditions become important. Current density has to be kept fairly high in order to bring down the time of electrolysis. Elevation of temperature greatly diminishes the chemical polarization of iron but the same applies to cementite. Chemical attack by the electrolyte also increases strongly with rising temperature. The latter has to be kept low, therefore, and the anodic polarization of iron has to be reduced by other means, such as presence of chloride ions stirring, frequent removal of carbide and possibly superimposed alternating current.<sup>7,8</sup>

The most effective stirring is rotation of the anode, preferably cylindrical, but this is usually not enough to remove the carbide particles. The specimen has to be scraped or at least knocked now and then. A brush in contact with the rotating specimen often gives poorer results, presumably

due to disaggregation of carbide, increasing its reactivity. If hydrochloric acid is employed as electrolyte, evolution of hydrogen occurs even at the anode and aids in loosening the carbide. In this case the carbide ought to be better protected on the anode than out in the solution to begin with. In no case, however, must the carbide remain on the anode to form a thick layer since that causes the current density and anodic polarization to be greatly increased and then the carbide also will begin to dissolve.

Regarding the electrolyte there is a wide-spread erroneous theory for the choice of anion. Treje and Benedicks<sup>9,10</sup> used sodium citrate as an electrolyte for isolation of both slag inclusions and carbide. With this electrolyte alone they expected to get oxygen evolution at the anode. They therefore wanted to add another anion that might be discharged instead of oxygen. They considered the electrode potentials  $\text{SO}_4$  1.90,  $\text{Cl}$  1.34,  $\text{O}$  1.33,  $\text{Br}$  0.99 and  $\text{I}$  0.52 V and concluded from them that sulphates and chlorides could not be used as they would not exclude oxygen evolution. The only rational anions would be  $\text{Br}$  and  $\text{I}$  which are discharged more easily than oxygen.

As far as the author has been able to find there have been no objections to this theory but it has been widely spread in the literature<sup>3,11,12,13,14</sup> and has exerted a great influence. Electrolytes containing bromides and iodides have been employed in a great many investigations, particularly by German workers.

In reality, however, the quoted anion potentials have nothing to do with the dissolution of iron. The electrode potential of significance is that of iron itself,  $-0.44$  V. Discharge of hydroxyl ions or other anions will occur only at very high current densities if the iron salts formed at the anode are not removed quickly enough or if the anode becomes passivated. Chloride ions are a very good means for counteracting passivity and a chloride, such as hydrochloric acid or ammonium chloride, is therefore a very suitable electrolyte. There are no valid reasons for using

bromides or iodides. Furthermore, iodide solutions are easily decomposed under the formation of iodine which attacks the carbide.

To support their theory, Treje and Benedicks<sup>9</sup> carried out some potential measurements. In a  $\text{FeBr}_2$ -solution at  $2-4$  A/dm<sup>2</sup> they normally found anode potentials as high as  $0.70-0.79$  V, that is, somewhere between the electrode potentials of iodine and bromine. However, these values are erroneously calculated, since the authors failed to see that the anode potential was negative in relation to the calomel electrode and hence added  $0.286$  to the positive value of the bridge reading instead of to its negative value. Recalculated, the true values are  $-0.22$  to  $-0.13$  V. Similar results were obtained in a control measurement by the author. In German literature<sup>15,16</sup> there is a precept that the anode potential should be kept below  $+0.50$  V in order to exclude oxygen evolution. Presumably, this high limit originates from the erroneous figures referred to. As shown above, the anode potential need not and should not be higher than  $-0.3$  to  $-0.2$  V.

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# Effect of Prestraining Temperatures on the Recovery of Cold Worked Aluminum

T. E. TIETZ,\* R. A. ANDERSON\* and J. E. DORN,\* Member AIME

## Introduction

Recent investigations<sup>1,2,3,4</sup> have conclusively shown that the strain hardened state of metals depends upon the temperature and strain rate of prestraining as well as on the total plastic strain. A typical example of the effect of temperature of prestraining on the work hardening of metals is reproduced in Fig 1. When pure aluminum is strained at liquid nitrogen temperature the upper solid stress-strain curve (OFGH) is obtained. But when pure aluminum is prestrained to  $\epsilon_2 = 0.153$  at atmospheric temperature and the test is then completed at liquid nitrogen temperature, the curve OBCD results. Thus a prestrain  $\epsilon_2 = 0.153$  at atmospheric temperature gives a flow stress  $C$  when continued at liquid nitrogen temperature, whereas a higher flow stress  $G$  results when the specimen is strained exclusively at liquid nitrogen temperature to the same strain  $\epsilon_2$ . Consequently the metal strain hardens more rapidly at the lower temperature. Obviously the strain alone is not a measure of the strain hardened state in metals.

Inspection of Fig 1 reveals that the flow stress at  $C$  is identical with that at  $F$ . This fact suggests that a strain  $\epsilon_2 = 0.153$  at atmospheric temperature induces the same work hardened state as a strain  $\epsilon_1 = 0.058$  at liquid nitrogen temperature. But this concept of equivalent strains is also in error. If curve  $CD$  is shifted to the left so that point  $C$  falls on  $F$ , point  $D$  becomes  $D'$ . Inspection of the curves reveals that the rate of increase of stress with strain at  $C$  is greater than the rate of increase of stress with strain at  $F$  even though the flow stresses are identical. Consequently  $C$  and  $F$  do not represent

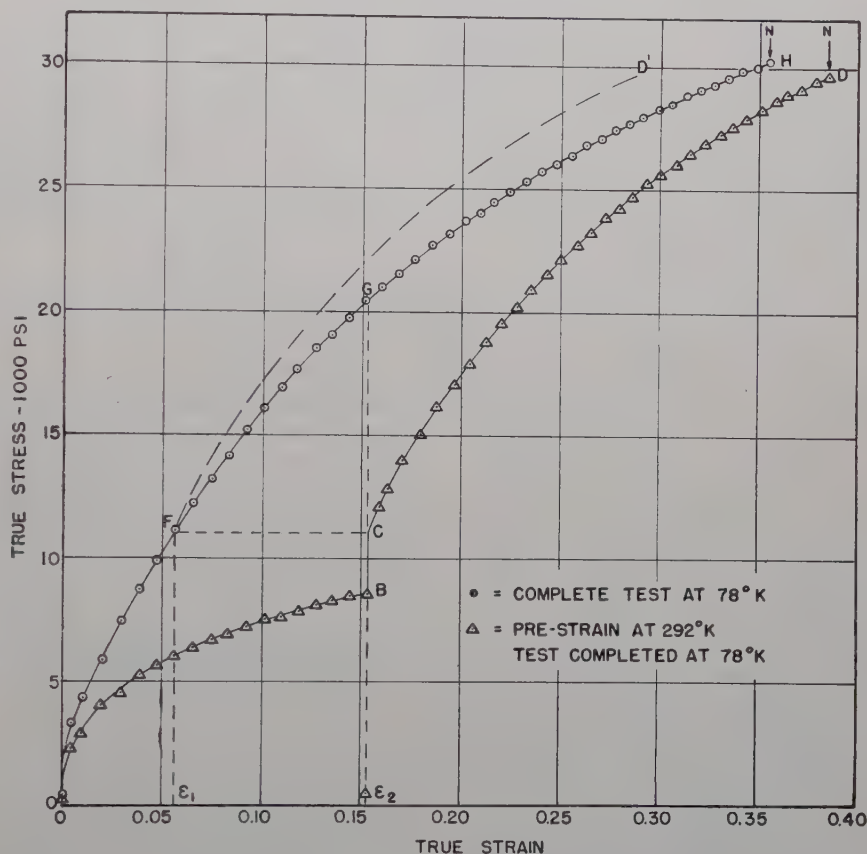


FIG 1—Effect of temperature of straining on the strain hardening of pure aluminum (99.987 pct).

identical work hardened states. It therefore becomes important to ascer-

tain how the strain hardened states at  $C$  and  $F$  might differ from each other.

In a more recent report<sup>4</sup> it was shown that straining at higher rates of strain increases the amount of strain hardening in a manner quite analogous to straining at lower temperatures. These observations suggested that the flow stress is not only a function of the strain but also a function of the strain rate-temperature history of straining, perhaps in accord with the Zener-Hollomon parameter  $\rho = \dot{\epsilon} e^{\frac{\Delta H}{RT}}$  (where

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<sup>1</sup> References are at the end of the paper.



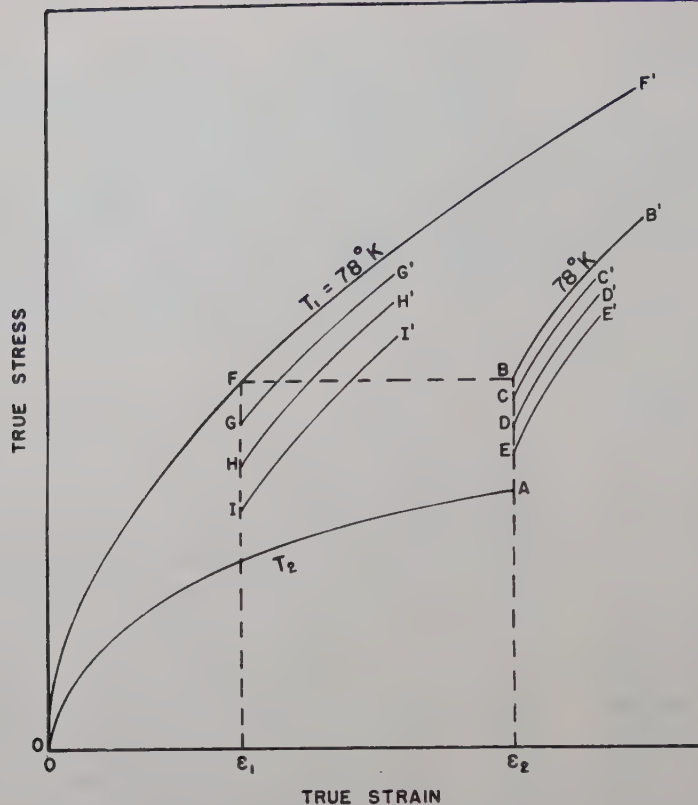


FIG 2—Method of evaluating the recovery.

$e$  = base, natural logarithm, and  $\frac{\Delta H}{RT}$  is an exponent of  $e$ ). If then a specimen is strained  $\epsilon_1$  at  $\rho_1$  and  $\epsilon_2$  at  $\rho_2$  etc., the flow stress might be given by

$$\sigma = \sigma(\epsilon_1, \rho_1; \epsilon_2, \rho_2; \dots)$$

Such concepts have practical utility for correlation and analyses of data but they fail to identify the fundamental causes for the effect of strain rate and temperature histories on the strain hardening of metals.

The well known effect of the strain hardened states of metals on their recovery rates suggested that a more complete identification of the effect of strain rate and temperature histories on the strain hardening of metals might be obtained from recovery tests. Preliminary data from two recovery tests<sup>4</sup> appeared to confirm this thought. The tests described in the following pages of this report were made in order to obtain more conclusive evidence on the effect of temperature of prestraining on the recovery of the flow stress.

## Materials and Testing Techniques

Pure aluminum (99.987 pct Al) was selected for these studies in order to minimize the effect of such factors as strain aging on the interpretation of

the data.

All of the tests were performed with equipment previously described in the literature.<sup>2,4</sup> Strains were measured to 0.001 and stresses were determined to about  $\pm 25$  psi. All recorded data are given in terms of true stresses and true plastic strains.

## Method of Evaluating the Recovery

The procedure used to determine the amount of recovery in this investigation is shown schematically in Fig 2. A specimen strained exclusively at liquid nitrogen temperature has the true stress-true plastic strain curve  $OFF'$  whereas a specimen strained to  $\epsilon_2$  at some higher temperature  $T_2$  and then immediately tested at liquid nitrogen temperature gives the curve  $OABB'$ . Thus a prestrain  $\epsilon_2$  at  $T_2$  gives the same flow stress ( $B$ ) at liquid nitrogen temperature as is obtained by straining exclusively at liquid nitrogen temperature to  $\epsilon_1$ . If the strain hardened states at  $B$  and  $F$  are identical the recovery rates of the two specimens should also be identical. But if the recovery rates are dissimilar the strain hardened states must be different even though the flow stresses are approximately the same at liquid nitrogen temperature.

The recovery of specimens prestrained to  $\epsilon_1$  at liquid nitrogen temperature was obtained by aging the specimens at some appropriate temperature for various times and then testing again at liquid nitrogen temperature. In this way a series of curves  $GG'$ ,  $HH'$ , etc. were obtained for the partially recovered material. For the data reported here these curves exhibited a sharp knee at the yield stress so that rather accurate determinations of the stresses  $G$ ,  $H$ ,  $I$ , etc. were obtainable by extrapolation to zero additional plastic strain upon restraining after recovery.

One complication arose in the analyses of the recovery data. Due to minor variations in properties from specimen to specimen, the flow stress  $F$  after prestraining to  $\epsilon_1$  at liquid nitrogen temperature was not quite the same for all specimens. The  $F$  value, however, was obtained for each specimen as well as the recovered stress, for example,  $G$ . The amount of recovery was then calculated as  $F-G$  for each specimen. In this way the effect of minor variations from specimen to specimen on the calculated amount of recovery was reduced to a negligibly small value.

A somewhat similar procedure was used to determine the recovery after prestraining an amount  $\epsilon_2$  at a temperature  $T_2$ . In order to evaluate the



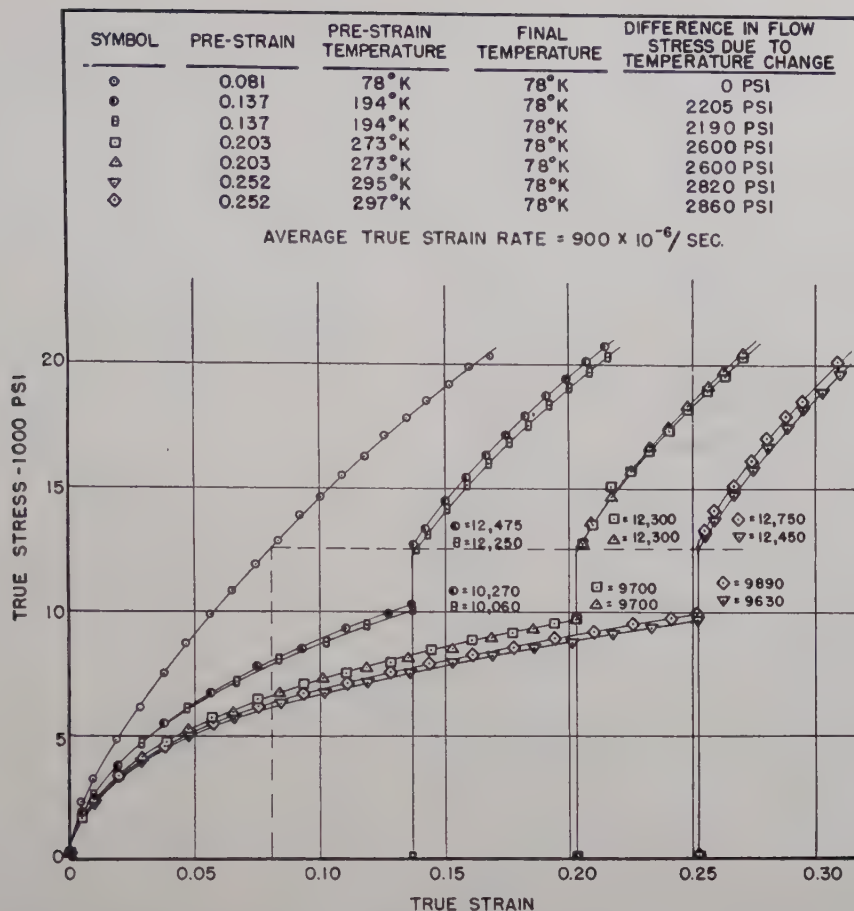


FIG 3—Effect of prestraining at various temperatures on the flow stress at liquid nitrogen temperature.

amount of recovery on a comparable basis, all tests after a recovery treatment were conducted at liquid nitrogen temperature, providing in this way curves  $CC'$ ,  $DD'$ , etc. The stress  $A$  differed slightly for each specimen strained to the same value  $\epsilon_2$  but stress  $B$  was found to vary in an analogous way so that  $B-A$  was substantially constant. This value was then used as a basis for calculating the amount of recovery. A specimen to be treated for recovery was strained to  $\epsilon_2$  and the stress  $A$  for the specimen was measured. After recovery the stress-strain curve  $CC'$  was obtained at liquid nitrogen temperature. In this way  $C-A$  was determined for the recovered specimen. The amount of recovery was then calculated as  $(B-A) - (C-A)$ .

## Experimental Data

The question of prime interest in the present study concerns possible differences in the work hardened state as revealed by recovery experiments after the metal has been subjected to various prestraining histories of such extent that the instantaneous flow stress is

constant. If different recovery rates are obtained for specimens that have been so prestrained that their flow stress at liquid nitrogen temperature is about 12,500 psi, it follows that the instantaneous flow stress under constant conditions of temperature and strain rate is not a measure of the work hardened state. Under such conditions the relative rates of recovery for the various prestraining treatments will serve to qualify the existing differences in the work hardened states that were generated. In order to permit this evaluation the metal must be prestrained just that amount at various temperatures to give a constant flow stress (12,500 psi in the following tests) when the test is continued at liquid nitrogen temperature. The conditions of prestraining used to accomplish this are shown in Fig 3. Prestraining to 0.252 at 296°K, 0.203 at 273°K, 0.137 at 194°K and 0.081 at 78°K gave approximately the same flow stress of 12,500 psi when the temperature was changed rapidly from that for prestraining to liquid nitrogen. In all cases the transfer to liquid nitrogen temperature was accomplished in less than 15 sec in order to reduce the

amount of recovery that might have occurred during the transfer interval.

It is significant to note that the two specimens prestrained at atmospheric temperature (296°K) gave slightly different stress-strain curves, but that the differences between the flow stress at liquid nitrogen and room temperatures were  $12,750 - 9,890 = 2,860$  psi, and  $12,450 - 9,630 = 2,820$  psi respectively, which are identical within the limits of experimental error. Similarly the difference in flow stress at liquid nitrogen (78°K) and  $\text{CO}_2$ -acetone (194°K) for the two specimens prestrained at  $\text{CO}_2$ -acetone temperature  $12,475 - 10,270 = 2,205$  psi and  $12,250 - 10,060 = 2,190$  psi which is again within the limits of experimental error. These data suggest that the effects of sampling can be largely eliminated by the procedure previously described for calculating the amount of recovery.

The recovery data are recorded in Fig 4 and 5. The amount of recovery was determined by the decrease in the flow stress at liquid nitrogen temperature upon recovery as described previously. Although there is some scatter in the data, the results are nevertheless



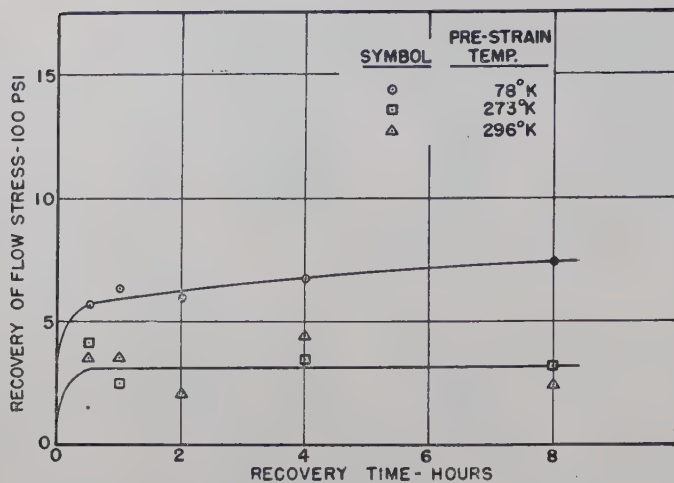


FIG 4—Effect of prestrain temperature on recovery at 273°K.

less conclusive. At 273°K the rates of recovery were relatively slow whereas satisfactorily higher recovery rates were obtained at 305°K. At both of these temperatures the specimens prestrained at 273 and 296°K presented approximately identical recovery rates indicating thereby almost identical work hardened states. But for both of the recovery temperatures used here the specimens that were prestrained at liquid nitrogen temperature (78°K) recovered most rapidly. Fig 6 shows the recovery data at 305°K on a log-log plot. As shown in Fig 4 and 5 the specimens prestrained at CO<sub>2</sub>-acetone temperature (194°K) recovered at an intermediate rate.

The data recorded in Fig 4 and 5 reveal that the high purity aluminum used in these tests recovers somewhat at atmospheric temperature after prestraining at atmospheric temperature. Such recovery must, therefore, also occur during the straining. It might, therefore, appear that the observed differences in the recovery rates after various prestrain histories is attributable to the possibility that recovery occurring during prestrain reduces the subsequent recovery for those specimens prestrained at the higher temperatures. In Fig 7 are shown recovery tests at the testing temperature for 273, 194, and 78°K. Although recovery does occur at 273°K in 1 hr, no recovery was detected in 4 hr at 194°K or in 1 hr at 78°K. Evidently the data obtained by prestraining at 78 and 194°K are not clouded by possible recovery during prestraining. Therefore, evidence is substantial that metal prestrained at the lower temperature recovers more rapidly than metal prestrained at a higher temperature even when the two conditions of prestrain-

ing result in the same instantaneous values of the flow stress at the lower temperature.

It is possible of course that greater rates of recovery are induced by the applied stresses during prestraining. In order to test this thought six recovery tests were made under stresses approximating the yield stress at the recovery temperature. No indication of a significant effect of stress on the rate of recovery was detected, thereby disqualifying this suggestion.

## Discussion of Results

The preceding experimental data reveal that high purity (99.987 pct Al) aluminum work hardens more rapidly for lower temperatures of prestraining. By appropriate selection of the amount of prestraining for each prestrain temperature it is possible to induce a work

hardened state that gives the same flow stress when straining is continued at liquid nitrogen temperature. In order to accomplish this, the specimens must be prestrained a greater amount for the higher prestraining temperatures. Although the instantaneous flow stresses at liquid nitrogen temperature are adjusted to the same value by this procedure, the rates of strain hardening differ systematically for the various prestraining temperatures. Specimens prestrained at the higher temperatures have higher rates of strain hardening for identical flow stress when they are subsequently tested at liquid nitrogen temperature. This fact suggests that although the flow stresses are the same, the work hardened states nevertheless differ in some yet obscure way as a result of the previous prestrain temperature history.

Specimens prestrained at the higher temperatures to a strain that induces a fixed flow stress at liquid nitrogen temperatures exhibit much slower rates of recovery of the flow stress upon storage at the same recovery temperature than specimens prestrained at lower temperatures. This not only occurs under conditions where the prestraining temperature is high enough to cause some recovery during prestraining, but it also occurs for prestraining temperatures at which the amount of recovery during prestraining is so small that it cannot be detected. The evidence is thus unmistakable that lower temperatures of prestraining to a fixed flow stress yield a more rapidly recoverable work hardened state. Consequently specimens of a metal so treated by various histories that they have the same in-

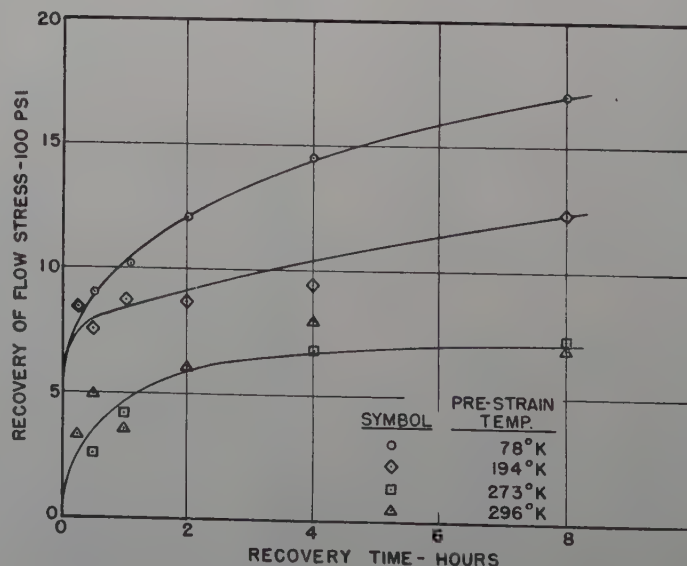


FIG 5—Effect of prestrain temperature on recovery at 305°K.



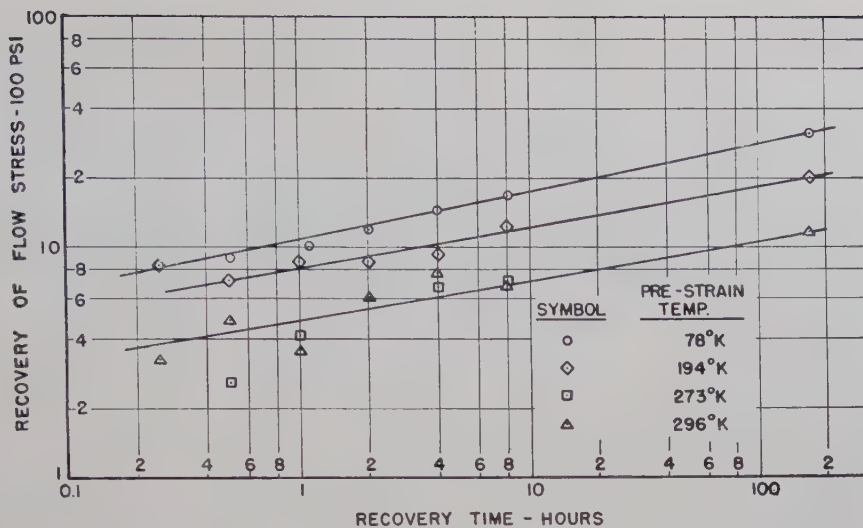


FIG 6—Effect of prestrain temperature on recovery at 305°K.

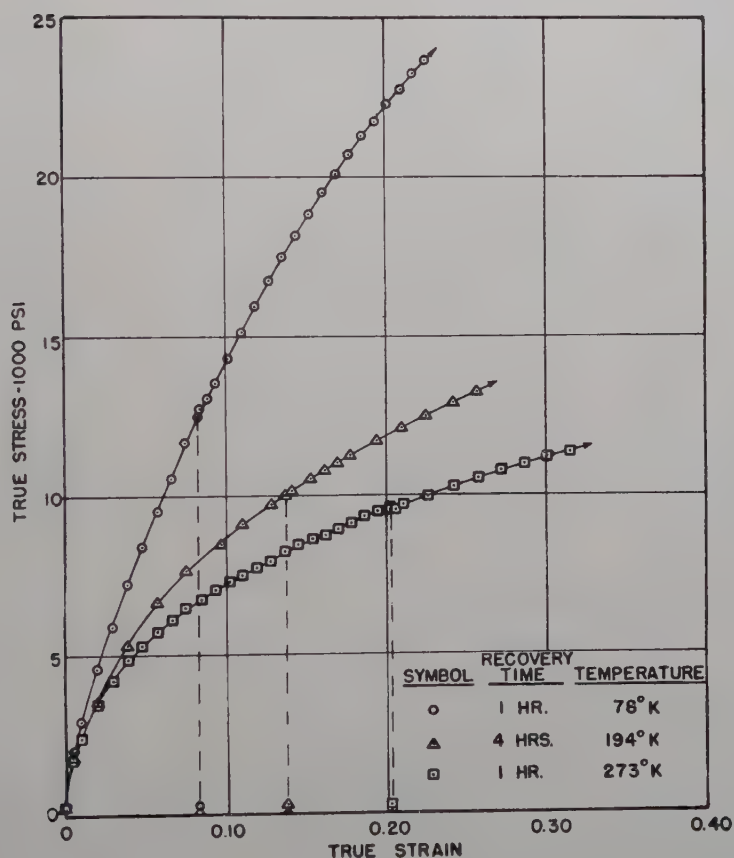


FIG 7—Recovery at testing temperature.

stantaneous flow stress under specified conditions of testing do not necessarily exhibit the same work hardened state.

The differences in these work hardened states is somewhat elucidated by their relative rates of recovery. It appears justifiable to assume that the work hardened state induced in cold worked metals is essentially attributa-

ble to imperfections of a type that are perhaps analogous to dislocations. The work hardened state is then determined by the size, distribution and density of the dislocations. Prestraining under different conditions of temperature, strain-rate and strain induced unique sizes, distributions and densities of the dislocations. Thus it is pos-

sible to acquire the same flow stress in a metal by various strain histories and yet have differences in the size, distribution and density of the dislocations. Upon recovery the more mobile dislocations will be activated and might migrate out of the mosaic blocks or be otherwise annihilated. The rate with which such a recovery process



would occur at a stated recovery temperature would depend upon the relative activation energies for the various sizes, distributions and densities of dislocations. The new work hardened state and the new flow stress following recovery would depend on the resulting dislocation statistics. Inasmuch as those specimens which were prestrained to a stated flow stress at the lower temperatures recovered most rapidly it appears logical to conclude that they also contain a larger number of more easily activated dislocations. Perhaps they contain a greater number of more readily activated dislocations more propitiously distributed as a result of the lower prestrain temperature to give nevertheless the same flow stress as is obtained by an appropriately larger prestrain at a higher temperature. Perhaps the dislocations generated at the lower prestrain temperatures are smaller, (as has been suggested on the basis of other evidence by Taylor<sup>5</sup> and Kauzmann<sup>6</sup> and Dushman<sup>7</sup>) but more numerous so that the same flow stress is achieved as is obtained by more extensive prestraining at the higher temperatures. The latter suggestion is of interest and will constitute the subject for further investigations.

## Conclusions

1. The mechanical properties of cold-worked metals depend not only on the instantaneous values of the

strain, strain rate and temperature but on the entire past history of temperature and strain rate during prestraining.

2. Neither the instantaneous value of the total strain nor the instantaneous value of the flow stress under stated conditions of strain rate and temperature are adequate for characterizing the strain hardened state.

3. Specimens of a metal can be so prestrained at various temperatures that the same flow stress is obtained when they are subsequently tested at the same instantaneous temperature and strain-rate. Those prestrained at the higher temperatures require greater amounts of prestrain.

4. Although such specimens have the same instantaneous flow stress, those prestrained at the higher temperatures have greater instantaneous rates of strain hardening.

5. The recovery rate of pure aluminum depends upon the temperature of prestrain; specimens prestrained at the lower temperatures exhibit much higher rates of recovery.

6. These observations appear to suggest that lower temperatures of prestraining induce the formation of smaller or otherwise more readily activated dislocations.

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## CORRECTION

M. K. Yen and W. R. Hibbard, Jr.: The Transverse Bending of Single Crystals of Aluminum. *Journal of Metals*, Oct. 1949, p. 716. Fig 16 and 17 are transposed.



# Flow and Fracture Characteristics of a Die Steel at High Hardness Levels\*

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## Introduction

Most structural parts which are heat treated are designed using strength properties which have been determined in the principal direction of the wrought material. For example, for rolled or drawn materials, properties are given for the rolling or drawing direction. The structures, however, may be loaded so that the critical stress is in some direction other than that for which the properties of the material are known.

Investigations of forged products,<sup>1,2,3</sup> have shown that while the yield strength and tensile strength of carbon steel billets and bars vary little with the direction of the test specimen in relation to the fiber, the contraction in area in tension tests and the impact strength in notched bar impact tests decrease in the transverse direction. The contraction in area and, consequently, the fracture stress of hard aluminum and magnesium alloy forgings have also been found to be lower in the transverse direction.

An investigation on aluminum alloy plate<sup>4</sup> likewise has shown the dependence of the fracturing characteristics upon the direction\* of the test specimens, the longitudinal direction being considerably stronger than the transverse direction and the normal direction, with the normal direction being the least strong.

The variation of properties with direction has been explained by a type of anisotropy called mechanical anisotropy. This anisotropy results from the elongation, in the direction of the principal strain, of certain phases, inclusions, and/or cavities in the metal

during working. A mechanical fibering is thus produced which seems to persist through annealing and heat treatment.

This investigation was initiated to determine the flow stress and fracture stress, at high hardness levels, at 90° to the rolling direction in a round steel bar. It is this direction which receives the critical stress in drawing dies machined from round bars. Preliminary tests showed a large difference in properties between the 0° and 90° directions. Consequently, it was felt that a more complete investigation, utilizing several types of tests, was warranted to determine the flow and fracture characteristics of a steel at various orientations, for a number of hardness levels. This investigation was conducted on an air hardening nondeforming die steel.

## Material and Procedure

The distribution of properties was

made on a 3-in. round bar of annealed high-carbon, high chromium steel of the following analysis:

	Pct
Carbon.....	1.53
Manganese.....	0.39
Silicon.....	0.27
Chromium.....	11.76
Vanadium.....	0.25
Molybdenum.....	0.81

The 3-in. bar was produced from an 8-in. ingot, which was annealed and forged to a 4-in. square billet. The billet was annealed and rolled to a 3-in. round which was then annealed and straightened.

This steel is an air hardening die steel which has very good dimensional stability on hardening; therefore, the residual stresses resulting from hardening would be expected to be low. A hardness survey across the diameter of the annealed bar showed no difference in hardness from the center to the outside. However, the test sections of all the specimens were taken approximately half way between the center of the bar and the surface to avoid any surface effect or possible porosity at the center.

Tension, compression and bend tests were made on specimens hardened and tempered at six different temperatures.

The tension test specimens, Fig 1, were machined from the bar at orientations of 0, 22.5, 45, 67.5 and 90° from the axis of the steel bar. The specimens were rough machined, heat treated and then ground to size. The test section on each specimen was lapped in a direction parallel to the axis of the specimen to remove any transverse scratches which might act as stress raisers. The specimens were tested in fixtures which insured concentricity of loading of less than 0.001 in.<sup>5</sup> The transverse strains were measured with a radial strain gauge,<sup>5</sup> the least count of which was 0.0001 in. change in diam.

The compression specimens, Fig 1, were machined from the bar at ori-

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1 References are at the end of the paper.

\* The direction of a test specimen can be defined as the relation of the axis of the specimen to a set of axes made up of the longitudinal, transverse, and normal directions in the material.



entations of 0° and 90° from the axis of the steel bar. The specimens were rough machined, heat-treated, and then ground to size. The specimens were tested between hardened ground steel blocks in a specially built ball-bearing die set, which assured pure compression. The ends of the specimen were lubricated with a drawing compound to minimize the friction. The transverse strain was measured with the same radial strain gauge which was used for the tension specimens. All the specimens were strained in excess of 4 pct (increase-in-area). No perceptible bending or barreling of the specimens was noted at these strains.

The bend test specimens, Fig 1, were machined from the bar at orientations of 0° and 90° from the axis of the bar. The specimens were rough machined, heat treated, and then ground to size. A photogrid, 100 lines per in.<sup>6</sup> was applied on the tension surface of the bend bar. Readings were made from this grid after fracture to give the strain at the middle of the tension surface. The strains were measured with a Filar eyepiece, using a gauge length of 0.01 in. Five gauge-lengths were meas-

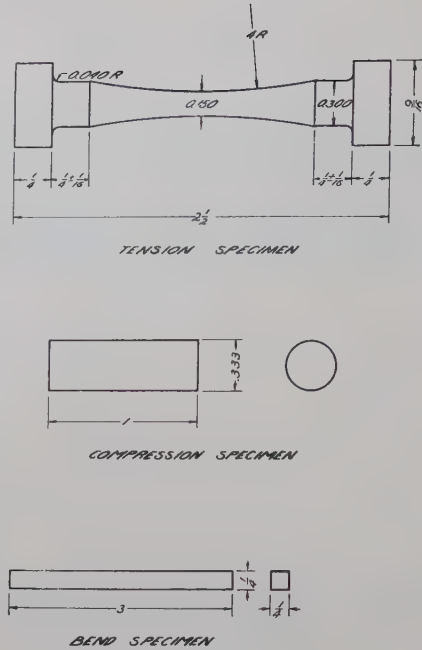


FIG 1—Test specimens.  
a. Tension specimen. b. Compression specimen.  
c. Bend specimen.

ured on each side of the fracture and the strain at the fracture was extra-

polated from these measurements. The specimens were bent in a “Wrap Former” around a 1.25 in. radius die.<sup>6</sup> All of the specimens fractured during testing at relatively small angles of bend. The bend angle was measured after fracture on a microprojector.

All of the test specimens for each tempering temperature were heat treated together, the specimens being held 40 min. at 1820°F, in a Vapo Carb Atmosphere furnace and cooled in still air. The specimens were tempered in a forced air convection furnace at 1000, 1025, 1050, 1100, 1150 and 1200°F for one hour.

The corresponding hardness values for the various tempering temperatures were as follows:

Tempering Temperature °F	Hardness Rockwell C
1000	62.5
1025	62.5
1050	59.
1100	54.5
1150	50.
1200	45.

## Results

### STRUCTURE

In order to show the disposition of

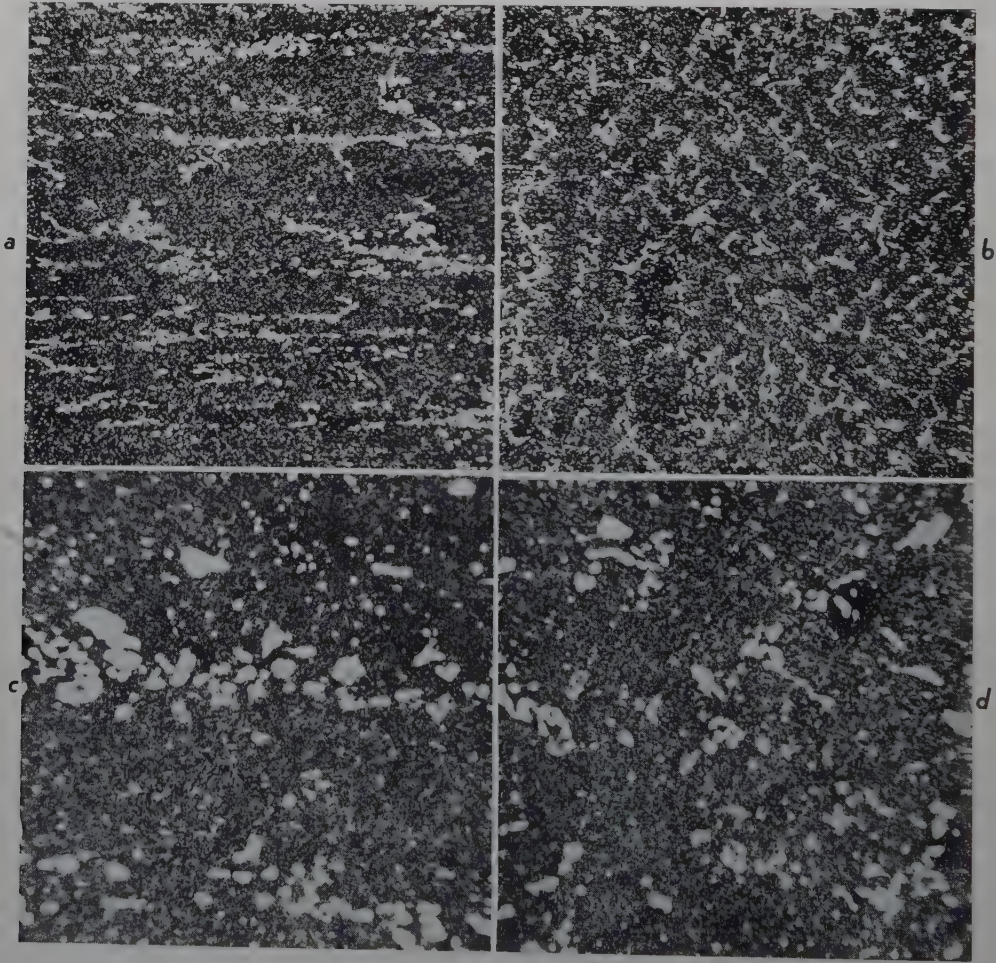


FIG 2—Structure of a 3-in. diam bar of die steel, quenched and tempered at 1200°F. Nital etch. a. Longitudinal direction. X 100. b. Transverse direction X 100. c. Longitudinal direction. X 500. d. Transverse direction. X 500.

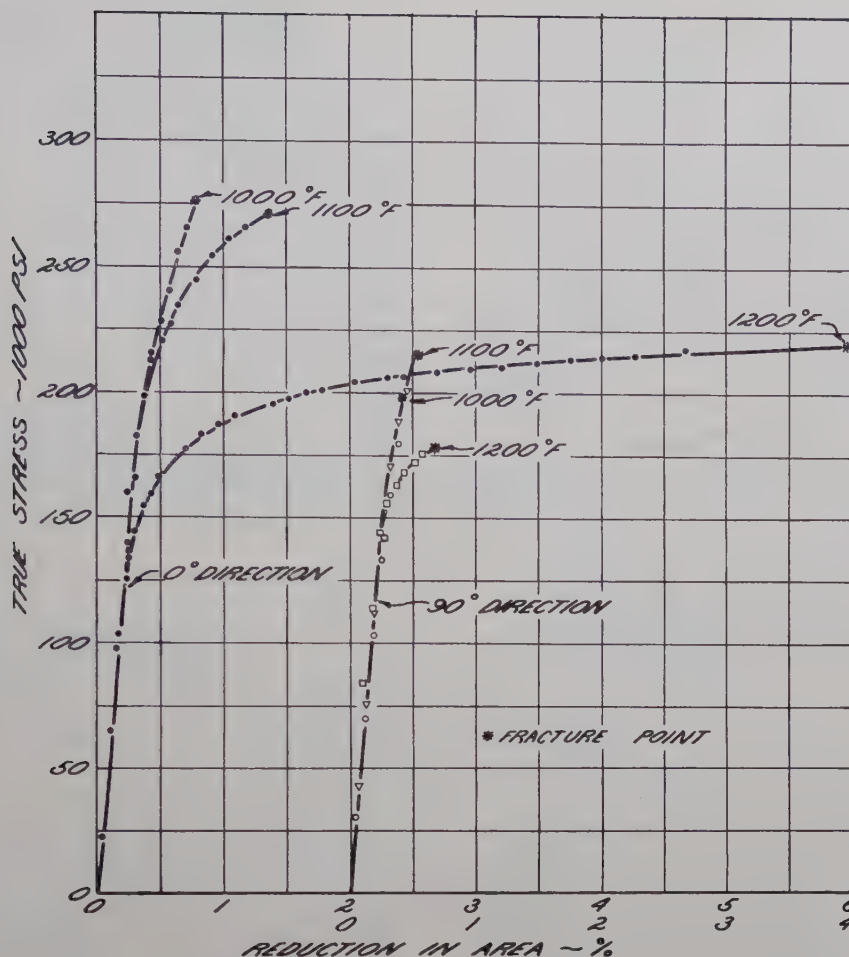


FIG 3—Typical stress-strain curves in tension for a die steel tempered at various temperatures and tested at several directions to the rolling direction.

the carbides, micrographs were made in the longitudinal and transverse directions of the bar in specimens which had been quenched and tempered at 1200°F. These micrographs are shown in Fig 2 at both 100 and 500 magnifications. It can be seen that the undissolved carbides have been strung out in the longitudinal direction of the bar by the fabricating operations.

#### TENSION TESTS

The results of the tension tests are shown in Fig 3, 4, 5, and 6. In Fig 3 a few typical tension curves are shown for the longitudinal and transverse directions at three different tempering temperatures. The fracture stress, ductility, and yield stress are shown in Fig 4, 5, and 6 as functions of tempering temperature and of specimen direction for three selected temperatures.

The fracture stress, Fig 4, for every orientation reached a maximum at a tempering temperature between 1025 and 1050°F. At 1000°F the fracture stress fell off for all of the orientations. The fracture stress at each tempering temperature decreased from a maxi-

mum at the 0° direction to a minimum at the 90° direction.

The reduction in area, Fig 5, was small for all of the tests but showed an increase with tempering temperature for all orientations. The increase in ductility was greater at each tempering temperature as the orientation changed from 90 to 0°. The yield stress, 0.2 pct offset, Fig 6, was the same for all orientations at each tempering temperature. The yield stress increased as the tempering temperature was lowered until a maximum was reached at 1025°F and then the yield fell off at 1000°F in a manner similar to the fracture stress.

#### COMPRESSION TESTS

Typical stress strain curves in compression are shown in Fig 7 for the longitudinal direction at three different tempering temperatures. The stresses in compression for 0.1, 0.2, 0.5, 1.0 and 2.0 pct plastic flow are shown in Fig 8. A small but consistent difference was noted between the orientations of 0° and 90°, the values for 0° always being higher. The values of yield stress in-

creased with decreasing tempering temperature and unlike the yield stress in tension, did not have a maximum at 1025°F.

#### BEND TESTS

The strains measured at the center of the tension surfaces and the angles of bend after fracture are given in Fig 9 and 10. Considerable difference is shown between the 0° and 90° orientations for both the bend strain and bend angle, this difference becoming larger with increasing tempering temperature. Both the strain and the angle increased with increasing tempering temperature for both 0° and 90° orientations.

#### Discussion of Results

A high degree of directionality was shown in the fracture stress and ductility for this material. At 1200°F tempering temperature the strength in the transverse direction was 18 pct less than the longitudinal direction and this difference increased at lower tempering temperatures. An even greater



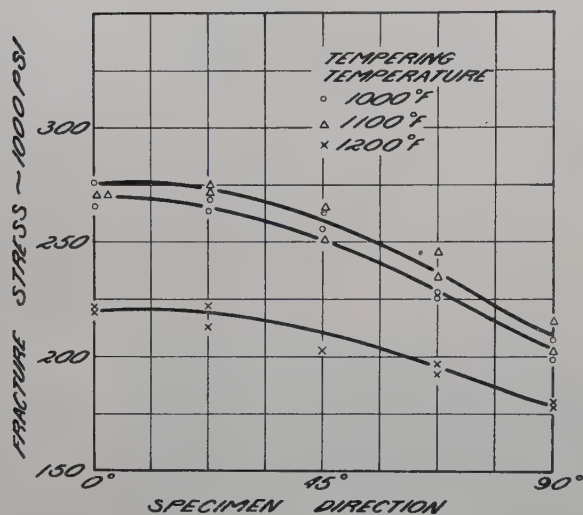
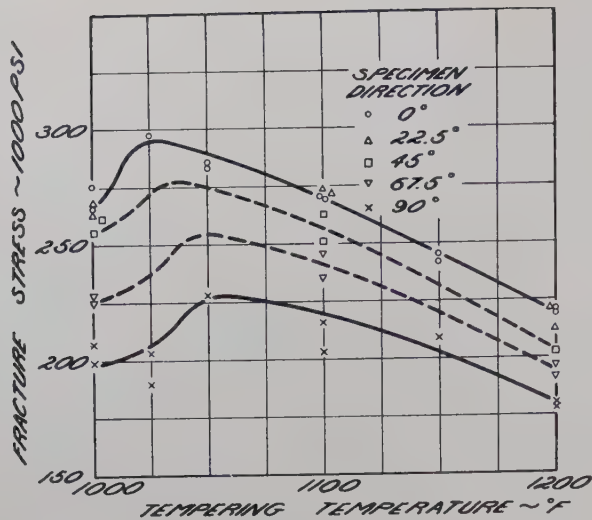


FIG 4—Dependence of fracture stress on tempering temperature and orientation.

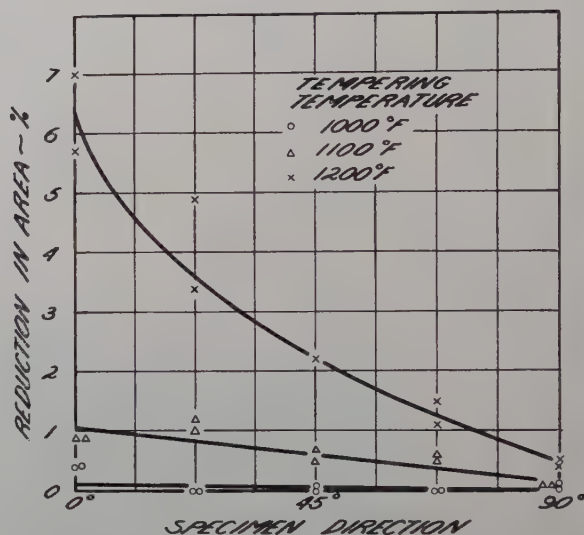
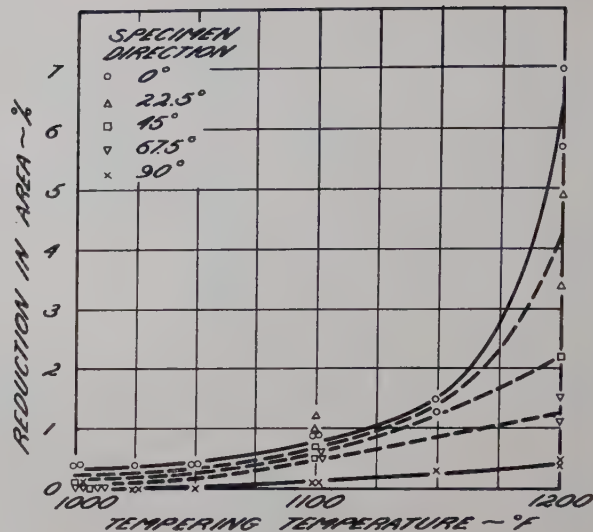


FIG 5—Dependence of ductility in tension on tempering temperature and orientation.

difference was shown by the ductility over the whole range of tempering temperatures used.

The decrease in fracture stress expressed as a percentage of the fracture stress at 0°, is shown in Fig 11 for the different orientations at three tempering temperatures. This decrement appears to be a function of the specimen direction and is also dependent upon the structure, particularly at 90°. This behavior is of the type which would be expected if discontinuities or cavities existed parallel to the axis of the bar. The yield stress in tension, however, showed no such directionality (the yield stress in compression showed a slight degree of directionality). This further supports the belief that mechanical anisotropy is the cause of the differences in fracture stress and ductility since a crystallographic preferred orientation normally causes differences in yield strength in various directions. Cubically aligned copper is

one exception, in that it shows little dependence of yield stress upon the direction of testing.<sup>7</sup>

The decrease in yield stress in the vicinity of 1000°F tempering temperature may be due to the retention of residual stresses which lower the apparent yield strength. The sharp drop in yield stress was not observed in compression. A comparison of the yield stress in tension with the yield stress in compression (0.2 pct), Fig 12, shows that between 1200 and 1100°F the yield stress in compression was slightly higher than that in tension. This is the usual case. However, below 1100°F, the tension yield stress decreased, whereas the compressive yield stress continued to increase.

The ductility of this steel is extremely low in the range of tempering temperatures used. On hardening, the steel retains a high percentage of

austenite (30 to 40 pct) which transforms on tempering.<sup>8</sup> A secondary hardening range is observed at tempering temperatures about 1000°F, Fig 2, where additional chromium carbides are formed. Consequently a mixed structure is obtained which is composed of variously tempered martensites, carbides, retained austenite, and possibly other transformation products. Small amounts of carbides, retained austenite or other transformation products would all cause a lowering of the ductility.

In the range of tempering temperature from 1000 to 1050°F the ductility is reduced to less than one per cent. Consequently, the fracture stress is lowered in this range.

The bend tests showed considerable difference between the 0° and 90° orientations when measured either by the strain or the bend angle. A comparison

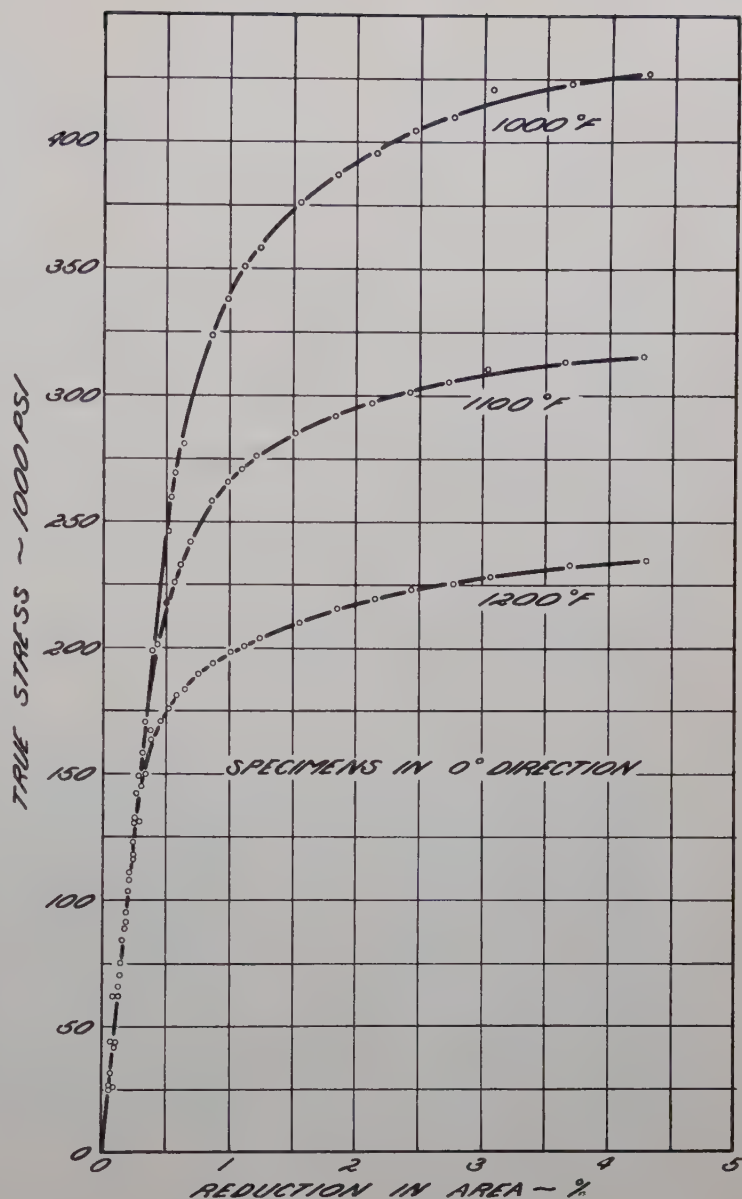
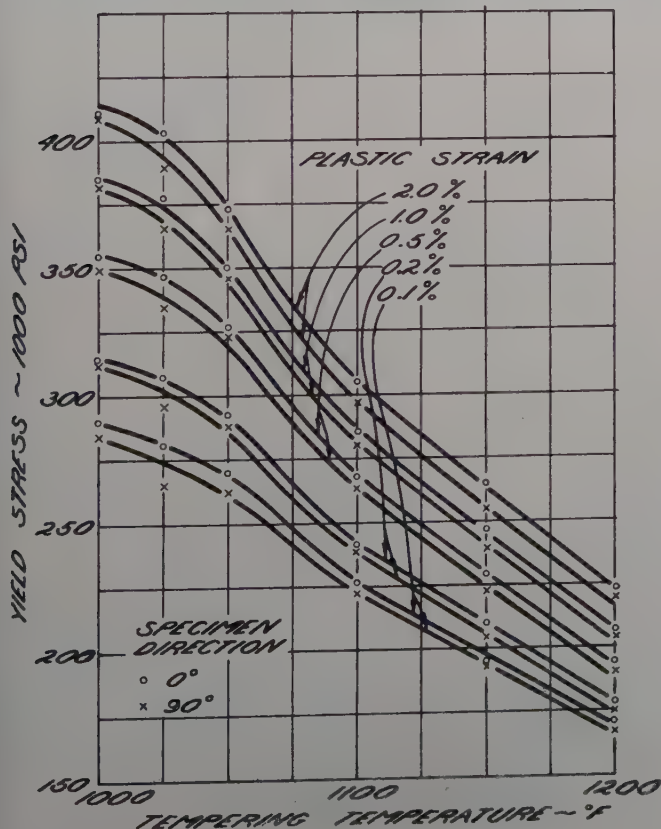
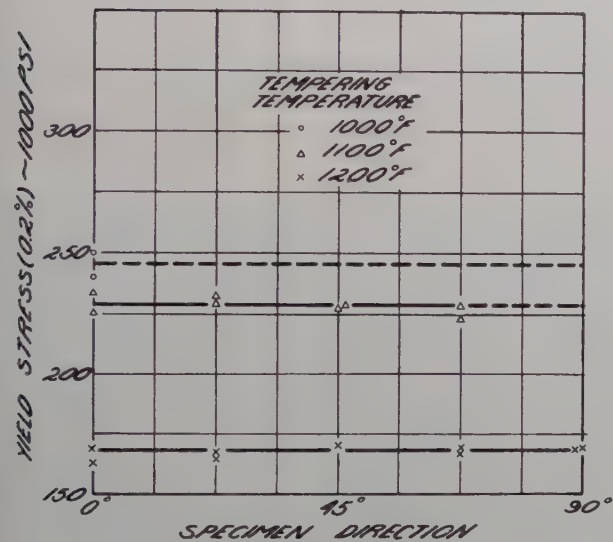
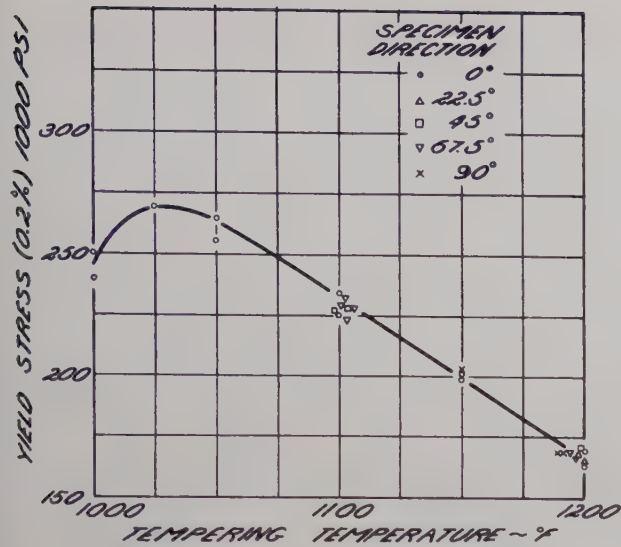


FIG 6 (Upper left)—Dependence of yield stress in tension on tempering temperature and orientation.

FIG 7 (Above)—Typical stress strain curves in compression for a die steel tempered at various temperatures and tested in the rolling direction.

FIG 8 (Lower left)—Dependence of yield stress in compression on tempering temperature and orientation for various amounts of strain.

of the strains measured in the bend tests with those in tension shows that the strains were greater for the bend tests although a slight degree of biaxiality\* exists at the center of the tension surface of a square bar.

## Conclusion

As a result of this investigation, the

\* On bending a square bar, a small transverse stress results at the center of the tension surface due to lateral restraint.<sup>6</sup>



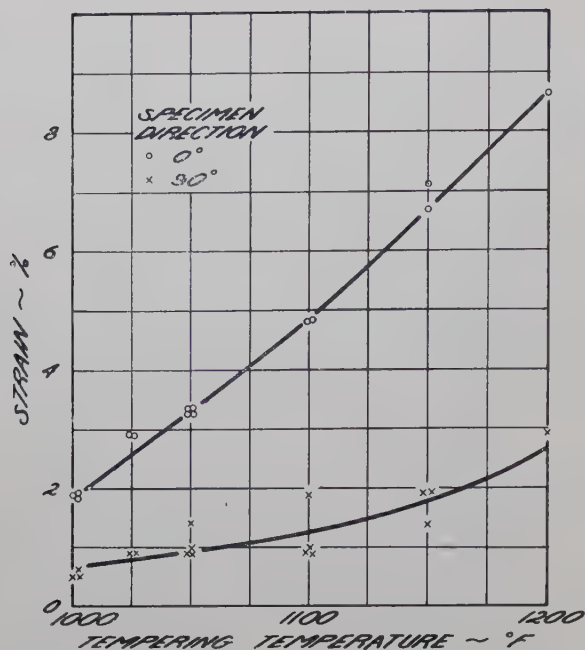


FIG 9—Dependence of bending strain on tempering temperature and orientation.

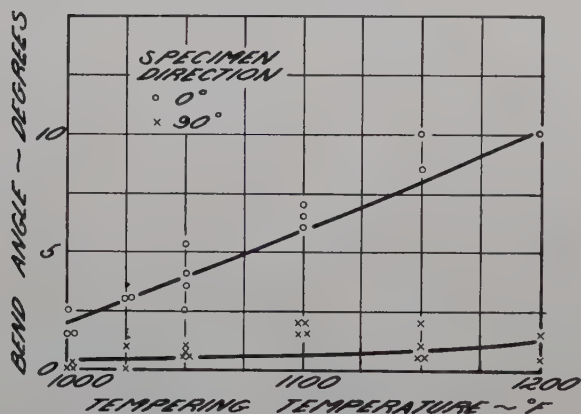


FIG 10—Dependence of bend angle on tempering temperature and orientation.

following conclusions can be drawn:

1. The fracture stress and ductility in tension and the ductility in the bend test exhibited pronounced directionality, properties always being higher in the longitudinal direction for every tempering temperature chosen.

2. The yield stress in tension did not vary with direction. The yield stress in compression varied only slightly with direction.

3. The directionality can be attributed to mechanical anisotropy caused by the carbide stringers developed in the fabrication of the material.

### Acknowledgment

The authors are indebted to the Office of Naval Research, U. S. Navy, for permission to publish this

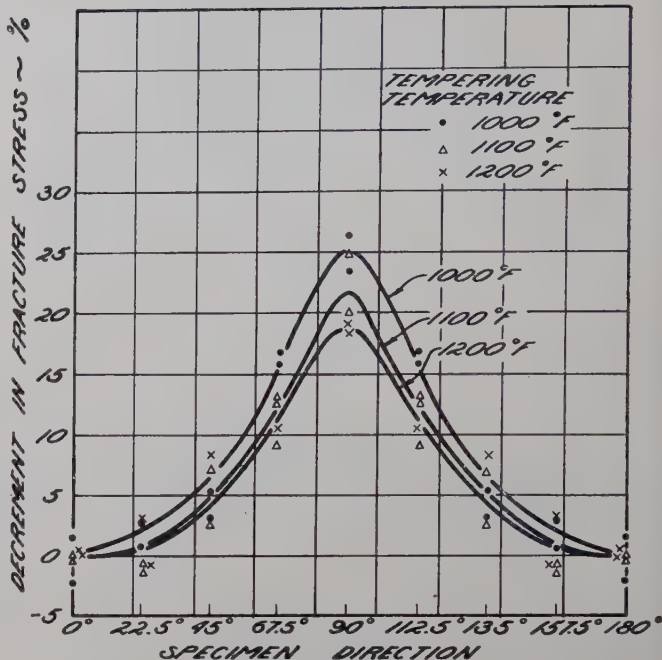


FIG 11—Decrement in fracture stress as a function of orientation.

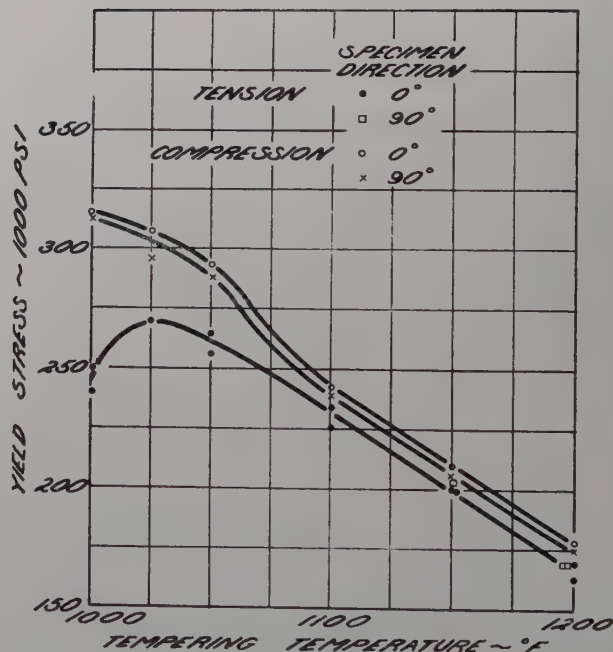


FIG 12—Comparison of yield stresses in tension and compression for 0.2 pct plastic strain.

information.

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# The Effect of Working and Heating Eutectic Structures

J. S. BROWN\* and A. G. GUY\*

With the exception of the work of Tammann and Hartmann,<sup>1</sup> no published information has been found on the structural changes produced in eutectic structures as the result of heating following plastic deformation. In part this lack of information may be due to the fact that many eutectic structures contain an intermetallic compound as a prominent phase, and as a consequence have been considered to be unworkable. For example, Tammann and Hartmann were able to obtain useful data only on those alloys that they could greatly reduce in thickness

cooling in the mold from about the eutectic temperature. The fairly coarse eutectic structure thus obtained was too brittle to work at room temperature without cracking. Fig 1b shows the worked structure obtained by compressing a  $\frac{1}{2}$ -in. cube 70 pct in about 15 sec at 550°F and then water quenching. The pronounced changes in the distribution of the two phases produced by heating the worked alloy at 850°F for 1 hr are shown in Fig 1c. Recrystallization of both phases appears to have taken place, and the mechanical properties of the cast alloy

tion, to have a granular structure after heat treatment: Sn-9 pct Zn (98 pct); Cd-82.5 pct Pb (60 pct); and Bi-43.5 pct Pb (90 pct). On the other hand the alloy Al-11.7 pct Si (60 pct) showed no change after heating. Tammann and Hartmann observed a distinct shrinking of the needles in unworked eutectics subjected to heat-treatment. In the present work inappreciable change in the eutectic structure was found on heating undeformed alloys.

In summary, it appears that eutectic structures can be recrystallized into a "spheroidized" condition by heating



FIG 1a—The cast structure of the Pb-33.2 pct Mg alloy; the Mg-Pb intermetallic compound appears dark and the Mg solid solution light.

Unetched. 1000 X.



FIG 1b—The Pb-33.2 pct Mg alloy reduced 70 pct by pressing at 550°F.

Unetched. 1000 X.



FIG 1c—The 70 pct reduced Pb-33.2 pct Mg alloy after heating for one hour at 850°F.

Unetched. 1500 X.

by cold rolling. This restriction placed on the investigation of alloys containing intermetallic compounds has been largely removed by the work of Savitskii<sup>2</sup> which showed that large deformations of such alloys can be produced by relatively slow compression at temperatures near the melting range. It was possible, therefore, to carry out the present work on a range of binary eutectics including one composed of two intermetallic compounds.

The essential results obtained on the alloy systems studied are illustrated by the Pb-Mg eutectic containing 33.2 pct Mg, Fig 1. The cast structure, Fig 1a, was obtained by melting together magnesium of 99.8 pct purity and chemically pure test lead, casting into a heated, small steel mold, and furnace

must have been significantly changed by the working and heat treatment.

Similar results were obtained with the Mg-59.5 pct Bi, Mg-36.4 pct Sn, Mg-65.4 pct Cu, and Bi-40 pct Cd eutectics, only the last of which could be worked at room temperature. Tammann and Hartmann reported a thickening and shortening of the needles of the eutectics which they heated about 10°C below the melting range for 1 hr after drastic cold rolling. They found the following eutectics, given the indicated amounts of reduc-

tion, near the eutectic temperature after severe deformation. If the eutectic contains one or more brittle phases the necessary deformation can be accomplished by pressing at high temperatures. It is reasonable to expect that this tendency towards equiaxed grains from the plate- or needle-like eutectic structure should be general since the energy relations and mode of growth in recrystallization are so different from those that hold for eutectic crystallization.

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Technical Note No. 27 E. Manuscript received July 27, 1949.

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<sup>1</sup> References are at the end of the note.



# The Fume and Dust Problem in Industry

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## Introduction

In this paper, as prepared for delivery at the Southern California regional meeting on Oct. 14, 1948, it was thought best to interpret the term "economics" in a rather broad manner and to include, in addition to the material losses and recoveries and associated monetary values (Part I), a limited discussion of the increased difficulties or the particular problem and the special requirements, as the particle sizes of the suspended particles range down from the relatively coarse to 100, to 10, to 1 micron or even to a fraction of one micron (Part II). Further, it is not quite in order to overlook entirely the community and individual health problems, although space requires the economics of this to be considered only very incompletely. Therefore, Part III, covering this phase of the subject, is very limited.

This paper, then, is divided into 5 parts or headings as follows: I Losses and/or values in suspended solids. II Particle size. III Dust and fumes in community and individual living. IV Means and Procedures for dust and fume collection. V Description or examples of specific equipment in service and of the several types used for dust and fume collection. Because of the wide extent and wealth of subject material available and the space and time limitation imposed, presentation and discussion are less than originally planned.

## I—Losses and/or Values in Suspended Solids

The weight involved in moving streams of industrial plant gases is commonly not appreciated, neither is their carrying power in the weight of solids maintained in suspension and

moved with the gas stream from a point of origin or pick-up to a point of dissipation or settlement. These, however, are major weight figures; for example, in a modern iron blast furnace there may be five tons of gas for every ton of iron produced and by the time this blast furnace gas has been burned in stoves or under boilers the weight of gas discharged to atmosphere is on the order of eight times the weight of iron produced. Similarly for nonferrous metallurgy there may readily be from 10 to 20 times the weight of gases discharged to atmosphere as there is metal produced.

A cement kiln in operation or a kiln in service to produce metallurgical lime may have on the order of 5 to 6 times the weight of stack gases as of clinker or lime produced, and at least the cement kiln, because of the very fine nature of its feed, is a very heavy dust producer.

It may be noted that there have been two developments in progress for nearly three decades. Both are extraordinary in the industrial economics effected and in their ready availability to ever larger units of operation and their ever widening importance in industry, and both are productive of great quantities of finely divided material in furnacing. The first of these is the flotation process for ores, especially the metallics such as copper, lead, and zinc; and the second, powdered fuel combustion for power plant, industrial plants and metallurgical operations. Today, new developments, for

example, flotation for the nonmetallics such as higher grade limestone for cement manufacture which requires still finer grinding and the powdered-coal-fired boilers with production ratings of over 1,000,000 lb of steam per hr, bring still more concentrated and hugely increased quantities of stack emission.

Perhaps the honors for the greatest interest in the quantities and values escaping in waste furnace and equipment gases belong to the nonferrous metallurgical operations. Their record of achievement in the installation of dust and fume collection equipment, largely baghouses or Cottrell electrical precipitators, is exceeded by no other industry.

Something of the magnitude and variety of equipment utilized in such recovery systems was covered by the writer in two papers presented to the Institute some 10 years ago.<sup>1,2</sup> It is not intended to repeat the material of those articles, but it is thought that they complement this offering and should be noted.

## COPPER ROASTERS

As the copper roasters are the first of the series of furnaces handling the copper-bearing concentrates in the usual copper smelter of today, it is in order to make them the first consideration.

Multiple hearth sulphide roasters, not hard driven, often maintain their dust loss through exit gases at 3 pct or below of feed to furnace; in hard-driven or maximum-driven furnaces, exit gas losses often approximate 7 pct of charge with a  $\pm 2$  pct variation for special conditions prevailing at some plants. A 5 pct loss of feed in a roaster gas exit, unless reclaimed, often makes the difference between a profit and loss operation, and in many cases substantial recovery is the very basis of dividend payments. As there is available very practical and successful equipment for the collection of the

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<sup>1</sup> References are at the end of the paper.

suspended solids in the acid-bearing copper roaster gases, all plants handling good tonnages of through-put material are equipped with some form of collection equipment; the most common and quite the most successful collection equipment makes use of high potential electrical discharge—the process of *Electrical Precipitation*, more commonly referred to as the Cottrell process. In general, the values in copper roaster feed are not confined to the copper only but include both gold and silver. Copper and gold show no appreciable fume production and their recovery in a normal roaster precipitator may be on the order of 10 pct higher than that of total solids (commonly there are present in the precipitate minor amounts of fumes and dust—as oxides and/or sulphates of lead, zinc, arsenic, antimony and the like) and 5 pct better than the silver recovery efficiency. Silver has a greater volatility than copper and gold, and particularly so in the presence of volatilized lead compounds.

No two copper smelters are alike as to losses with their flue gases, the variation being surprisingly large and usually excessive in hard-driven plants or older plants, or with poor maintenance as indicated by leaky flues and general large air infiltration conditions. It is, however, worthwhile to go through a sample set of figures of furnace losses and collections for a copper smelter and while the figures are not those of any one smelter they are more or less representative of all. Consider a custom smelter, as an example, with a charge of 1000 tons per day of crushed ore averaging 7 pct copper, and 1000 tons per day of concentrates averaging, say, 35 pct copper. The crushed ore which may be considered coarse in comparison with concentrates, is fine when considering only run-of-mine ore. However, even on their crushed ore, say,  $\frac{1}{8}$  to  $\frac{1}{4}$  in. dimensions, there is much decrepitation and production of fines in the actual furnacing operation, so that it will be productive of finely divided particles readily carried off by the furnace-exit gas stream. Nevertheless, the major dust will be derived from the concentrate fraction of furnace feed, and the collection in a precipitator will approximate 15 to 20 pct copper, say, 17½ pct as an average. On flotation concentrates alone the dust loss may reach 10 pct of feed. On crushed ore with appreciable decrepitation taking place, dust loss is often 5 pct and if we say 7½ pct as an average dust loss and a precipitator

recovery of 98 pct on the copper, the distribution of copper may be placed as follows: In new feed to roaster 420 tons copper, in Cottrell collection 73.5 tons, in stack gases 1.5 tons. The latter, the stack loss, is 0.36 pct of the copper in furnace feed and it is very evident that there is the desirability of high efficiency of recovery. With copper at a market of 23¢ per lb (1948) even the recovery of 98 pct and the accompanying loss of 1½ tons of metallic copper, or roughly  $\frac{1}{3}$  of 1 pct, is an item of some interest and will bear further consideration.

## REVERBERATORY FURNACES

In the past, the operation of reverberatory furnaces has varied so widely from plant to plant and from time to time in the same plant, that the majority of operators have considered their waste-heat boiler installation as a good enough collector and have disregarded further dust and fume-collecting equipment.

Reverberatory practice, however, is divided into two types, first with all feed passing through the multiple-hearth roaster, and second, that type in which the wet concentrate is directly fed to the reverberatory furnace. As is to be expected, the latter type of operation uses more fuel and has a greatly increased gas volume. Both these conditions are normally productive of a higher dust loss from furnace. In addition, however, the wet feed, although preventing dusting at charge time, is often after an interval of time productive of disruptive surface conditions due to the very rapid change of water particles to steam vapor, and this effect is productive of dusting.

A reverberatory, with roasters pre-treating its feed and working on a mixture of crushed ore and fine concentrates, is considered operating as well as may be reasonably expected if its stack loss is on the order of  $\frac{1}{2}$  to  $\frac{1}{4}$  of 1 pct of furnace feed in copper—about the same fraction of a percent loss in gold, and 1 to 1+ pct in silver—the latter increasing with the presence of lead fume in the gases. On the basis of a smelter operation with new copper-bearing feed of 1000 tons per day, 20 pct copper, 0.3 oz gold and 2 oz silver and present-day market (1948) of 23¢ per lb for copper, such reverberatory loss is considerable, amounting to approximately \$250.00 per day. However, 85 pct of the total value is in the copper and since the present price is nearly twice that of war-time fixed

price, it does not follow that such losses would warrant the immediate construction of high efficiency and relatively expensive recovery equipment. Perhaps it is in order to consider means for a relatively lower-efficiency mechanical recovery.

Where the reverberatories are directly fed with undried and unroasted concentrates the losses in the exit gases after boilers are appreciably greater (commonly double or even more) than stated above for calcine-fed reverberatories, and the dust collection is justified. Commonly such investment where the copper content in the feed is high, is returned within a very few years of service of collecting equipment by the value of recovery.

## COPPER CONVERTERS

Generally, modern and large converters will lose from the furnace, with their exit gases, on the order of 1 to 2 tons copper per day and this may be 1 to 3 pct of the copper produced. It varies widely from plant to plant due to grade of matte, type of flux and procedure of operation.

The actual solids lost, both dust and fume, may be several times the copper loss and in lead and zinc-bearing mattes the elimination of these two constituents as oxides or sulphate compounds is almost 100 pct with the gases.

Because of the necessities of operation there is always a tremendous infiltration of outside air at the converter hoods, and thus a volume in the flue just back of the converter stands may be on the order of 100,000 to 150,000 cfm for a present-day large converter. Such excessively large gas volume, in proportion to air supplied to converter at tuyeres or to tonnage of material treated, or to weight and nature of suspended solids in the exit flue gases, makes the economic handling of converter losses a rather uncertain problem. A portion, approximately one half the total suspended solids, is relatively coarse particles, being essentially the material expelled mechanically by the effects of converter blast, and this is substantially collected in a flue or settling chamber. The remaining fraction, however, is normally very fine and it may justify only a mechanical collector, say, with 80 to 90 pct (on copper) efficiency or it may justify an electrical precipitator of about 98 pct efficiency and perhaps, with the latter, at least twice as expensive to build.

Roughly, one can say the North



American capacity for production of copper (in copper reduction works) and in some 30 plants, approximates 1,000,000 tons per year, or at \$400.00 per ton copper, \$400 000,000.00. Certainly this is a primary sizable, and for the security and good of the country, a most important industry! On the basis of a furnace loss of 7 pct of the copper content and a recovery from the furnace gases of 96½ pct of the furnace loss there is a copper recovery in flue dust of around 67,550 tons copper, or expressed in dollars at \$400.00 per ton a total of approximately \$27,000,000.00 with a final loss from stack of about ¼ of 1 pct of original plant input of new copper-bearing material. These are, of course, quite generalized figures but show the magnitude and ability of our copper smelter metallurgists to confine their stack losses to an exceedingly small minimum through the installation and skilled use of a large variety of dust collecting equipment. The main dependence, however, is on electrical precipitation for the collection in acid-carrying gases and of the exceedingly large amount of fines which the modern flotation milling plant provides.

#### DIFFERENTIATION OF DUSTS, FUMES, AGGLOMERATES, ETC.

As a distinction between recoveries of copper values and those of lead and zinc in exit furnace gases, it is to be noted the copper is recovered almost entirely as a "dust," while in the lead and zinc suspensions both dust and fume are present, with the fume often the largest fraction. We define a dust particle as substantially a fragmental particle derived by percussion or crushing as the ore and fuel and flux are processed and handled through mines, mills and furnaces. It commonly has a chemical and mineralogical composition approaching that of the furnace feed. In practice the major fraction of the dust is said to average 5 to 50 microns, but blasting and crushing operations always produce a minor fraction below the 3 or 5 micron range and a minute portion may be not over or even less than 1 micron.

A very special suspension product results from solid carbonaceous fuel combustion. This runs in composition from the raw coal and partly burned particles of fuel to tar fogs, soot, soot agglomerates, fragmented ash particles and the fused, rounded and commonly hollow particles, the so-called "fly ash" of powdered-fuel suspension combustion. The fly ash particles cover a fair

range in size but a goodly portion are on the order of 1 micron, and broken sphere fragments less than a micron. Other than the tar fogs they are all solids and do not represent a volatilized product but no doubt some of the finer carbon or soot particles, before agglomeration has taken place, are products of decomposition, probably carrying with them traces of actual tar, and may be looked upon for size and function, too, as "fume"-like or "fog" particles.

Fumes, as compared to dust, are constituents or compounds as of or from the charge, vaporized in the hottest areas of the furnaces and chilled to a solid form out of contact with walls. These fumes bear no physical resemblance to the new materials of furnace feed, often existing in the flue gas suspensions as an "igneous concentration" product. They are generally assumed to be of rounded form and of exceedingly small diameter, from a few hundredths of a micron to one micron in diameter or occasionally somewhat larger. There is always an appreciable agglomeration present, probably due to the effects of electrical charges on the solid particles occurring in fume-laden gases. Similar agglomeration effects are to be noted in liquid suspended particles, that is, in mists and fogs. Examples of the latter are tar and acid fog. The effect of agglomeration or of agglomeration and coalescence is to produce fewer and larger particles and thus increase the settlement rate.

#### LEAD AND ZINC FURNACING UNITS

Lead and zinc reduction works, in the roasting and reduction departments, produce both dust and fume, perhaps to the gross amount of 5 to 10 pct of the new metal-bearing material to reduction works, but thanks to modern development in dust and fume-collecting equipment, including both baghouses and electrical precipitators, actual stack losses in modern lead smelters and zinc works of the electrolytic type, including the roasting or sintering operations essential thereto, may be on the order of a small fraction of 1 pct of new metal input. In fact these enterprises today rarely are in trouble from dust-fall, but doubtless some who do not convert their SO<sub>2</sub> production into acid, or liquid SO<sub>2</sub> or elemental sulphur, do have what is termed an SO<sub>2</sub> problem.

#### IRON AND STEEL WORKS

Our main mineral wealth producers here in the West have, in the past,

been our lead, zinc and copper enterprises and therefore we have been particularly interested in their dust problems. However, we have, within recent years, developed several plants in ferrous or iron metallurgy and these plants have dust problems, quite as great in volume and weight as copper and lead smelter enterprises, but with values in the collected products for which most iron smelter men are entirely unwilling to set any worthwhile figure. Nevertheless, modern iron and steel plants are eager to clean their combustible gases from blast furnaces and coke ovens and public officials look with concern at open hearth stacks and sintering machine stacks with large and objectionable "plumes."

There are three main sources of dust-bearing furnace gases passing to atmosphere in an iron and steel works, first the iron blast furnace, second the sintering machine and third, the open hearth reverberatories. In plants of a generation ago, there were only the crudest, if any, dust and fume-collecting devices on blast furnace equipment. From the stack discharge gases from the blast furnaces, the stoves and adjacent boiler plants burning the blast furnace gas, there was almost a rain of reddish colored dust, giving the blast furnace a most undesirable reputation. Today, however, all modern plants realize the savings and benefits of clean gas in stoves and boilers, and even greater economic advantages are secured in plant operation by burning the low Btu blast furnace gas under coke ovens, and the high Btu gas from coke ovens in domestic or steel plant operation. For by-product coke oven service the blast furnace gas must be practically as clean as outside air, the usual specification calling for not over 0.002 grains of solids per cf gas calculated at N.T.P. Almost equally important is clean gas for modern closely spaced checkerwork of the blast furnace stoves, but here a specification not quite as rigid as considered necessary for use in coke oven combustion, is permissible and the usual figure is stated as not over 0.007 grain per cf as calculated to N.T.P. conditions. To accomplish the degree of cleaning, the usual current design calls for (1) a dry dust settler adjacent to the blast furnace providing a 180° turn for the gases, in other words, an impingement type dry cleaner; (2) such dry dust collector to be followed by an efficient wet scrubber, usually counter-current type in baffle or tile packed chamber; (3) the scrubber to be followed by a



wet or film-type electrical precipitator. The dry dust collector may have an inlet concentration of 10 to 15 grains per cf, an outlet of 3 to 5 grains per cf, the scrubber with an inlet which is the outlet of the dry dust catcher and an outlet of 0.25 to 0.3 grains per cf and lastly an electrical precipitator with an inlet concentration of the scrubber outlet and precipitator outlet concentration of 0.007 grains per cf. These correspond to average efficiency for each piece of series-operating equipment and as developed only on its own average inlet and outlet concentration, of approximately 70 pct for the dry dust collector, 94 pct for the scrubber and 97 pct for the precipitator. The precipitator collection is substantially all fumes, largely those of the alkali or alkali-earth compounds but occasionally fumes of zinc are present.

The economic results of this train of dust and fume collection equipment are very considerable indeed. Clean gases to stoves mean nearly theoretical combustion, with high heat; no suspended solids in gases permits close packing of brick work and greatly increases the storage and release of heat, and the absence of any fine deposits upon the checker work permits rapid accumulation of heat and its equally ready release. Thus the furnace is better served and responds by greatly increased uniformity of operation, by increased daily production or greater capacity and by simplicity and ease of handling. These are not readily-figured benefits over one day's operation, but over several years provide noteworthy economics of operation and the maximum of production.

The numerous reheating and soaking pits of a steel works do not commonly discharge visible or appreciable quantities of suspended solids but the sintering plants and open hearth furnaces are at times producers of considerable clouds of dust with some fume. These pieces of plant equipment, in their stack discharge, reflect the nature of the dry feed to the furnace; where there is much fines there is corresponding furnace loss, where there is fume-making material (for instance, galvanized iron scrap) a fume discharge will be evident.

The open hearth reverberatories, in particular the newer and larger units, say 100 to 150 tons of steel production per charge and above, are now very generally being equipped with waste heat boilers. Such boiler installations in themselves are fair dust collectors and provide the very decided additional

advantage of cooling the furnace exit gas volume and thereby correspondingly decreasing the gas volume to be handled, so that additional collection means after the boilers are relatively easily supplied and installed. In reverberatory furnaces where blast furnace pig iron is a major constituent of the feed it is also common practice to add iron ore as a means of effecting the combustion of the carbon of the pig iron. At the time of feeding that charge to the reverberatory and perhaps for an hour or more thereafter, there will be heavy reddish stack discharge. This is not a serious loss as regards weight of materials or of values at the furnace but it is certainly one of the items that give the steel works an evil name as a maker of community dust. Reverberatory furnaces, depending upon their size, condition of feed or of fineness and fuming characteristics, and degree of "hard driving," range in dust loss from around 2 tons per day in a small hearth to around 10 tons of furnace dust loss per day in a very large furnace. As in a great many cement plants, it has been the practice to provide one chimney for each furnace, and this practice adds to the cost and inconvenience of any form of dust collecting devices. Once the procedure is adopted of installing waste heat boilers on each reverberatory and bringing all the boiler exit gases to a central point for treatment, the cost and feasibility of dust and fume collection installation will be greatly simplified, which makes for the minimum of expense. It is evident that one central recovery plant will cost less and be easier to handle than several individual dust recovery units.

The sintering machines in use at iron works are of comparatively recent introduction. The usual type is commonly provided with a large diameter, horizontal type of cyclone which serves to collect a coarse constituent of suspended material passing the grates; but in general, when fine ore or flue dust constitutes a major element of the sinter feed, there is a nuisance stack loss if not an important material loss. This equipment will receive due attention for dust collection along with the reverberatory department if and when the collection question is not one of plant economy but one of community betterment as regards appearance, cleanliness, saving in wear and tear, and the like. The installation of collecting equipment is neither more nor less pressing than in many other industrial operations or plants, such as cement plants, modern powdered-fuel-

fired power plants, railroad yards, handling equipment and the like.

## CEMENT PLANTS

Cement plants are large producers of dusts and fumes in their furnace gases, as would readily be expected from the nature of their operation. This operation includes preparation of a furnace feed of exceedingly finely ground limestone and clay or shale, 86 to 90 pct or above of minus 200 mesh (a 74 micron particle size just passes a 200 mesh screen), and the furnacing of this mixture to a point and temperature sufficient to produce a sinter. Commonly the prevailing temperature in the clinkering zone of the kiln is in the range of 2500 to 2700°F. On the basis of 600 lb of raw mix to 1 bbl finished cement, dust losses in stack discharges have been reported all the way from 10 to 120 lb per bbl. Needless to say the latter is abnormal, and good plant operation will not long permit such a loss. However, 30 to 40 lb per bbl kiln loss is quite ordinary and at 40 lb per bbl and 5,000 bbl per day the kiln dust loss (not stack loss) per day is 100 tons.

In former days of cement plant operation, before government regulations became the dominating and costly practice they are today for all of us, manufacturers were disposed to figure the value of their clinker so low that they were reluctant to give worthwhile values to the recovered kiln discharge dust. Today, however, with high labor and operation costs and still higher prices for cement, the dust becomes of considerable value to the producer—such figures of \$1.00 to \$2.00 per ton, occasionally more, being the practical basis of consideration. Since the U. S. production of Portland cement for 1947 is given as 185 million bbls it is evident we are talking of a kiln dust loss of over 3 million tons per year. It is to be understood that not all this dust reaches the atmosphere and is distributed around the surrounding community as a rain of dust, for the great majority of cement plants have some provision for collection. Their dust-collecting equipment runs all the way from an enlarged kiln-end housing or a boiler setting to an electrical precipitator, and includes baghouses or settling chambers, scrubbing systems, or one large or several small cyclones. On the whole, good collection service is provided by the industry. Possibly over half the total dust produced is collected—between one and two million tons per year.



## POWER PLANTS

A field somewhat similar to that of cement manufacture as far as large gas volumes and dust loadings are concerned, is that of the new powdered fuel-fired-boiler power plants. Units in this field are ever growing larger and boilers producing 1,000,000 lb of steam per hr, are being built with corresponding gas volumes of around one half million cfm at the average prevailing temperature of about 300°F. In the earlier powdered-fuel power plants and even in some modern ones upwards of 90 pct of the ash in the coal is discharged with the stack gases. The practice of burning coal in suspension has permitted the use of coal, without combustion difficulties, much higher in ash content than formerly was permissible. This, no doubt, along with the tremendous size of the modern power plant, has led to the very general complaint of the discharge of chimney solids (fly ash). As an example, take a power plant of a million kw capacity burning 12,000 tons of coal per day of possibly 12 pct ash, and assume 75 pct of ash in chimney discharge and not the 90 pct which has been occasionally reported. There results a stack loss of fly ash, so named, of approximately 1000 tons per day. Part of such stack loss is excessively fine, with many 1 micron particles, and usually  $\frac{1}{2}$  of the total dust particles are less than 10 microns in diameter. While American practice has favored the dry collection and predominantly that by the Cottrell process of electrical precipitation, the English have leaned toward the scrubber types with neutralization of the acidity resulting from sulphur dioxide collection.

In a British power plant<sup>3</sup> which has been reported in successful operation with better than 95 pct removal of dust and sulphurous acid from plant gases, the neutralization has been effected by use of a mixture of lime and limestone so that the active neutralization agent is reported as the bicarbonate of lime. This operation of neutralization, they state, is accomplished without the building of caked material, or scale (which we assume is a bisulphite salt) of the usual and very serious insoluble and voluminous calcium deposits so commonly encountered in non-precisely controlled scrubbing of waste gases containing SO<sub>2</sub> with suspended lime or limestone in the scrubbing liquor. Past experience in attempts to scrub the dust and sulphur-bearing compounds from cement plant gases does, indeed, make one most hesitant to accept with-

out thorough proof any scrubbing scheme for sulphur-bearing dust-carrying gases in which the neutralization is to be accomplished with a lime compound, and where the assumption or the assurance is given that no serious build-up of lime-carrying deposits will occur.

All indications point to more severe restrictions as to stack emanations from power plants and city factories for the future, and it is well if it is so understood by technical staff and plant management and plans and equipment are provided to accomplish the desired end. A statement was made some years ago by an English investigator that the damage resulting to structures, furnishings, clothing and health of the people was not less than  $\frac{3}{8}$  shillings for every ton of raw coal burned. Since at that time  $\frac{3}{8}$  shillings probably equalled about 55¢ of our money, it is easy to figure raw coal combustion without treatment of discharge combustion gases as running to hundreds of millions of dollars. The damage and nuisance and unpleasant results of raw untreated coal combustion are, of course, open to a wide range of interpretation, evaluation and even argument and one whose interests are normally technical is well advised to be cautious and conservative in his figures and remarks.

## GENERAL

In the preceding account there has been covered in a brief and general discussion the losses of wastes from stacks and chimneys of metallurgical operation of industrial and power plants. Stack losses for the entire United States, without the incorporation of present-day quality and recovery equipment of industry, smelting and power plants, would easily reach a billion dollars or more per year in values. With suitable equipment installed to collect suspended solids from furnace and industry exit gases, community savings of the above amount may be effected. Nevertheless, for health, for community, for good housekeeping and for the acceptance of the responsibility to be a credit to the community, which every industrial plant really plans to provide, there are required still further effort, expansion and investment for suppression or collection of vent or stack emission.

## II—Particle Size

If one would understand the problem of handling, controlling and collecting suspended particles in gases,—aerosols

as they have been termed—it is necessary to have a mental picture of the relative sizes and the special characteristics that go with such range of particle sizes. Over the years various relationships have been developed by one observer after another and these scattered data have been gathered into a very useful chart (Fig 1) by C. E. Miller and published in *Chemical and Metallurgical Engineering* in March, 1938 (p. 113). Due to the large amount of convenient and related information, this is reproduced here for convenience and by the courtesy of the publisher, McGraw-Hill Co.

It is of interest to point out several of the interesting features of this chart compilation. Reference to the lower left hand corner shows the relationship of standard screen size as compared to micron sizes. The particles just passing a 325 mesh screen are approximately 44 microns in diameter and while this is about as fine as one cares to go with screen sizes it is to be noted that 44 micron is still in the first third of size range given and such 325 screen, passing a 44 micron particle, is just about midway point or the sizes of many industrial operations, for example, sulphide ore, flotation pulps, pulverized coal, combustion ash (flyash) cement, and others. It will be noted few dusts go as low as 1 micron but many fumes and mists (including fogs) go down to about 0.02 micron, for instance, zinc oxide fume, tobacco smoke, and oil smoke. A half micron particle is nearing the edge of visibility with the most powerful optical microscopes and at 0.1 micron diameter particles show Browning movement and thereby display no tendency to settle.

To place the respective diameters and rates of fall in figures more common to our experience, Table 1 taken from Gibbs<sup>4</sup> and here somewhat extended to express the rate of fall both in foot per second and miles per hour is presented.

**Table 1 . . . Particle Diameter vs. Rate of Fall (Spherical and Still Air)**

Diameter of Particles		Mi-crons	Rate of Settling		
Screen Size*	Cm		Cm per Sec	Fpm	Miles per hr
150†	10 <sup>-2</sup>	100	30	59.0	6.711
	10 <sup>-3</sup>	10	0.3	0.6	0.06711
	10 <sup>-4</sup>	1	0.003	0.006	0.00067
	10 <sup>-5</sup>	0.1	0.00003	0.00006	0.0000067

\* Tyler Standard screen size, 200 mesh = 74 micron; 325 mesh = 44 micron.

† Approximate, size of opening in mesh to pass 100 micron (spherical) particle.



It is obvious from Table 1 that a mild breeze, of about 5 miles per hr, would permit settlement of only 100 micron particles or those rounded particles passing a 150 mesh screen; and for 10 microns diam and below, no settlement should be expected in the ordinary always-moving air currents about us. It is therefore a welcome and well-known phenomenon that atmospheric particles below settlement size are continually agglomerating into larger particles and in due course of time do settle. Observing the ordinary Tyndall beam and the huge mass of floating particles observable therein it is a satisfaction to realize that there is not a continuously increasing density of particles and that normally the Tyndall beam reflects an equilibrium condition wherein about as many old particles are agglomerated and removed by settlement as there are new particles entering the beam. The dust particles usually seen in a Tyndall beam are from 1 micron downward and that fraction from about 1/2 micron downward is invisible individually. However, when sufficiently concentrated into masses, the appearance is that of a cloud.

Two types of suspended particles are of particular interest to those whose activities in mining and smelting operations are in the West, and in the non-ferrous field. The first of these is silica dust, which in a certain size range has a health hazard. On the Miller chart the range is given as from 10 microns with a lower limit somewhat indefinite, as there is a question mark showing at 0.25 micron; others give the danger size range as from 5 micron to 1/2 micron. More particular mention will be made of this under a later division of this paper. The second type of suspended particle is white SO<sub>3</sub>, (or SO<sub>3</sub> · xH<sub>2</sub>O) a so-called "fume," but in actuality a fog or mist that escapes in such tremendous volumes particularly from our copper plant stacks. On Miller's chart the diameters of these SO<sub>3</sub> particles are given for the range of approximately 3 microns down to 1/2 micron. One series of interesting investigations of size of fumes for a number of products such as ammonium chloride, sulphur trioxide, phosphorus pentoxide, sodium peroxide and the like, were carried out about 25 years ago by H. Remy<sup>6</sup> in Germany. He followed the size change from formation until it was "aged," that is, agglomeration took place. He found these fume products aged very slowly after formation about 1/2 micron in size and that on "aging," that is agglomerating and

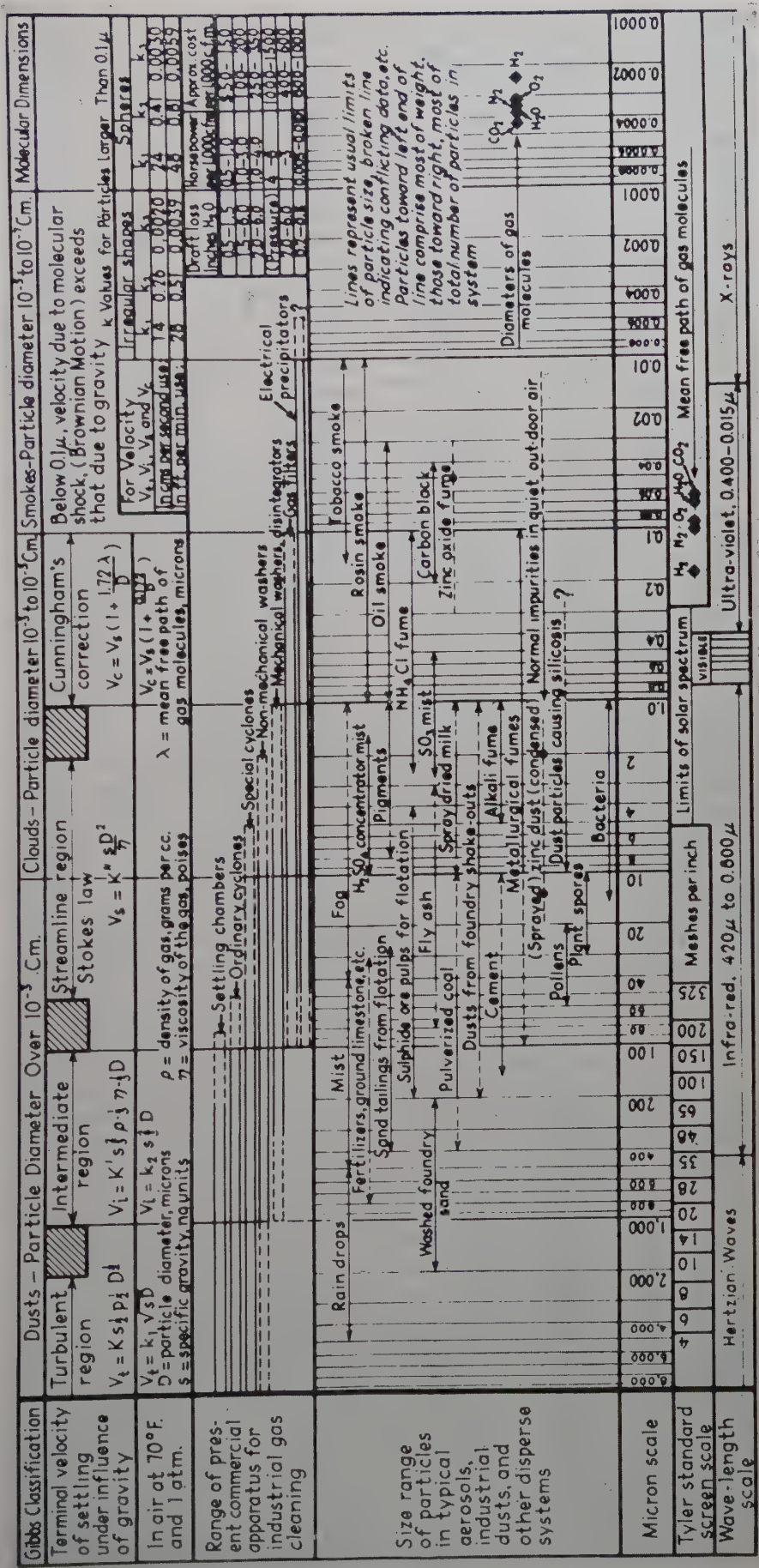


FIG 1—Summary of properties of some typical aerols. Size relationship to physical characteristics.  
Compiled by C. E. Miller, *Chem. and Met. Eng.* (1938) 45, 133. (Courtesy Chemical and Metallurgical Engineering.)



settling, they were somewhat over 1 micron in size. It is to be interpreted that if the settling distance had been that of many feet instead of the inches of his glass vessel, the agglomerates would have still further increased in size to the upper limits we often detect on a slide of settled fume, that is, to 3 microns or more.

### III—Dust and Fumes in Community and Individual Living

One who worked around our Western mines and lead smelters about 40 years ago noted a general indifference to possible health dangers. There were cases of silicosis on the one hand and lead poisoning on the other, occasionally both in one individual. Similar plant visits today make one realize the great advances that have been made in today's practice. Nevertheless, we still have a long way to go to reach desirable working conditions that are free of health hazards. Much of the betterment has come from understanding the necessity of proper ventilation and the installation and operation of proper equipment plus the equally vital understanding that cleanliness is an important consideration. Particularly around lead mines and smelting enterprises it is essential that simple health measures be enforced, such as the washing of hands before eating, the shower bath at the end of a shift, and changing and washing of underwear and work clothing. The reduction of incidence of lead poisoning by such measures is striking, and one has only to remember the poorly-kept plant of earlier years and compare it with an intelligently operated mine or plant of today to realize the change.

The average productivity per man hour in a well-run lead smelter or lead mine or in the handling and treatment of relatively high silica carrying ores, is more than sufficient to cover the costs of good working conditions, and in not a few places makes the difference between a profitable and nonprofitable operation.

Where it is a matter of health hazard there is the greatest anxiety to be certain and precise on the atmospheric content and character and state of subdivision of the suspended solids. In spite of a vast amount of endeavor, the development of many types of equipment and practices and procedures for particle counting and particle-collection weighing, there has resulted no one

method of procedure or equipment which may be called standard. Dust and fumes differ enormously in their collection characteristics, and while one type of equipment may give the most consistent and most accurate analysis of dust content in a given gas, it by no means follows that it will serve with equal fidelity on another type of suspended solid, or in a greatly changed set of conditions even with the original suspended solids. Therefore it may be said that there are some uncertainties still existing in reporting suspended solid concentration by any of the half dozen or more widely used types of apparatus.

Added to the problem of equipment is that of whether the basis should be expressed as the number of particles in a given gas volume or the weight of the suspended solids. The problem may be visualized by considering 1 cm cube of quartz and subdividing it into cubes of 1 micron edge length. The original 1 cm cube has 6 sq cm of total area while the equivalent mass in 1 micron cube has 6 sq meters and the number of micron particles becomes  $10^{12}$  or a million million. On the basis of 100 million particles per cf of air the 1 cc of the original quartz would be dispersed in an air volume of 10,000 cf.

Health authorities in investigating the hazards of occupation have leaned to the consideration of a count of number of particles rather than weight on the basis that the relatively coarse particles and that portion of the fines below about  $\frac{1}{2}$  micron are not relatively reactive to lung tissue; the coarse, because the moist and hair-like lining of the air passages collects and removes them, and the very fine ( $\frac{1}{2}$  micron and below) because they substantially conform to the characteristics of gases and move in and out of the lungs without chemical action with the tissues.

In a practical sort of understanding the particle counters and the weight followers have agreed that 1 mg of crushed quartz material contains about 300 million particles.<sup>8</sup>

The count-the-particle investigators are largely responsible for our laws on permissible concentrations of dust particles per cubic foot of air and such specifications are for particles which are less than 10 microns in diam as counted on microscopic fields. For example, the State of Illinois<sup>6</sup> through its health department specified permissible limits of dust by count as follows:

Total dust—50 million particles per

cf of air.

Free silica—5 million particles per cf of air.

Asbestos—5 million particles per cf of air.

Silicates—5 million particles per cf of air.

Alundum—15 million particles per cf of air.

Carborundum—15 million particles per cf of air.

Particles counted in microscopic field are limited to the all minus 10 micron sizes. Immediately after firing a blast to break rock in almost any hard rock mine a dust count will show about 50 million particles per 1 cf and it is usually necessary to reduce this to around 10 million particles in order to keep to 5 million or under of silica. Such a specification as this involves extended studies in mine ventilation. Allowable settling periods, use of sprays, bags, filters and the like all help to reduce the particle concentration to an acceptable figure. It seems the old days of one shift a day at a mine, blasting at end of shift and allowing 8 to 16 hr for workings to clear of dust particles resulting from the use of high explosives, are passing, and in our large low grade underground operations it is customary to work around the clock. Whereas in the old days a full shift was required to move the broken rock, substantially all by hand power, nowadays with a modern mucking machine the broken rock is removed in a couple of hours. Equally reduced is the drilling cycle and thus the opportunity for natural settling of rock particles is greatly lessened in comparison with the old days, and an acute problem in maintenance of limited dust concentration confronts the modern operator of larger tonnage in the lower grade ore mines.

### IV—Means or Procedures for Dust and Fume Collection. Both Test or Laboratory Equipment and Commercial Installation in Mine, Smelter, Power-house and Factories

The type of equipment effective for collection in commercial operations varies with the fineness of the suspended particles as will be noted by reference again to the Miller chart. That chart indicates 5 fundamental types of equipment, viz, (1) settling chambers (including wire-hung dust chambers), (2) Inertial or cyclonic

equipment (including baffled chambers and many types and kinds of cyclone collectors), (3) Spray chambers and/or scrubbers, (4) Filters (baghouses), (5) Electrical precipitators. A 6th, however, is on its way, industrially, and that is supersonic precipitation. For test work, little use is made of cyclone equipment if very high or complete collection is the end object, but the other four procedures mentioned are all used in test and laboratory work and in a large number of modifications. Two additional procedures have had considerable study: (1) thermal precipitation, and (2) impingement on a prepared surface with the particular object of counting the particles as they appear on a glass slide under suitable magnification. Impingement on liquid wet wall surfaces in a bath is also used for weight samples by health authorities.

Due to the protracted discussion required for any worthwhile review of the test methods proposed and used, which would unduly lengthen this paper, it is felt those particularly interested in this field should consult the extensive literature available.<sup>7,8,9,10</sup>

However, it is worth our while to consider some cost figures in commercial operations and respective efficiencies of the several types of collection equipment. You will note on the Miller chart several small tables on the right giving draft loss, power requirements and cost per 1000 cf of gas treated and, except for the cost item which needs to be doubled or more for today's installation, the data are quite in line.

It is cheapest to collect only the coarsest dust and here, if we can afford to pass everything on the order of 100 micrograms and finer, a settling chamber is the most economic type of equipment. However, you will note from the Miller chart that 100 microns or 150 mesh is less than halfway along the range of usually suspended dusts in the majority of industries. Note, for example, sulphide ore pulps from flotation, foundry dusts, cement, fly ash and the like. Of course such settling chambers have low draft losses and corresponding requirements for power are small (commonly a stack is sufficient) but, if a fan is required, it may be for ½ in. or less of draft loss and require ½ hp or less per 1000 cf of gas treated. As far as the writer knows, no enterprise having a modern dust problem has attempted to solve it by settling chambers during the last 25 years or thereabouts. Such settling chambers as are used today are inci-

dental—for example, a particularly large flue originally constructed and in anticipation of greatly increased plant size. One device, making use of the fact that decreasing the distance of fall increases collection, proposes a chamber with large number of superimposed shelves spaced a few inches apart—the Howard collector. At one time it was of particular interest to acid manufacturers using pyrites to prevent pyrites cinder from reaching the Glover tower. However, the restricted spacing between plates resulted in much trouble from choking of passageways so that gradually their use was abandoned.

Quite the contrary development has taken place with regard to inertial or cyclonic type of equipment. Special designs and modifications, almost without limit, have appeared during the last 15 to 20 years and many of these have added to the efficiency and range of fineness susceptible to collection by centrifugal equipment. Probably the most notable advance has been the recognition of the importance of decreasing the diameter for increasing efficiency and offsetting the capacity loss per item of equipment by using a multiplicity of cyclone units instead of one on a given gas volume. A number of investigators have reported such studies in various technical society proceedings or in the technical press, to which those desiring more details should refer.<sup>11,12,13</sup> Some of these special cyclones do good work on particle sizes appreciably below 10 microns, although there is also an escape of 10 microns and minus 10 micron particles. The basis of these newer studies on effect on recovery are well illustrated by a table prepared by Evald Anderson<sup>14</sup> which sets up the increased gravitational pull as the radius of collector is decreased from a 10 ft radius to a 1.5 in. radius while maintaining, through all changes in radius, a 60 fps tangential velocity (Table 2).

**Table 2 . . . The Centrifugal Separating Force in a Circular Gas Stream with a 60 fps Tangential Velocity for Different Radii, Expressed in Terms of Gravity, that is, Gravity = 1 in.**

Radius of Curvature of Gas Path	Separation Force
Gravity.....	1
10 ft.....	11 × gravity
5 ft.....	22 × gravity
2.5 ft.....	45 × gravity
1.0 ft.....	112 × gravity
6 in.....	224 × gravity
4 in.....	336 × gravity
2.5 in.....	518 × gravity
1.5 in.....	896 × gravity

The above appears almost too good, but as a practical consideration there

must be taken into consideration the turbulence existing at the interface of the stationary cylindrical wall and the high-velocity centrifugally-moving gas stream. This is of considerable magnitude and lowers the collection or separating efficiency. Pressure drop is an important consideration on all centrifugal equipment and in general, at least up to 6 or 8 in., efficiency is improved by increasing pressure drop. Commonly such equipment runs at pressure drops of 1½ to 6 in., and doubling the cost figure given by Miller for 1000 cf of capacity is in order, viz, \$200.00 to \$400.00 per M. of gas treated.

**SCRUBBERS**

Of extraordinary extent of field of application, varying from simple to complex design, and from small to extraordinary large power requirements are *spray chambers* and *scrubbers*.

Their service is, of course, limited to materials or products which it is permissible to wet and collect as a sludge, and if ordinary steel is to be the structural material, then only to that type of gases with their suspended solids which are not acid, or if acid then with a continuous addition of neutralization agent as a protection against corrosion. The simplest of these starts with a spray in a flue or a stream of water running over grid or lattice work and ends at the combined cyclone-scrubber construction of Theisen,<sup>15</sup> the Pease-Anthony<sup>16</sup> and the very recent Venturi scrubber.<sup>17</sup> Both the Theisen and the Venturi scrubber perform excellent service on very fine materials such as the fines present with the dusts and fumes from black liquor furnace combustion of kraft paper mills, the fumes and fine dusts of open hearth steel manufacture, iron blast furnace gas and the like. Their handicap is the large power requirement to drive the special-bladed Theisen scrubber runner, or the power required to provide the pressure drop and high gas velocity in the throat of the Venturi scrubber.

It will be noted in the Miller chart that the "Mechanical Washer, Disintegrators" cover the Theisen, and the latter is often known as a "disintegrator." The Theisen has an efficiency of recovery practically to the 1 micron size and it will also be noted the power requirements are on the order of 4-6 hp hr per 1000 cf. Many of the old Theisen washer-disintegrators handling iron blast furnace gas required power on the order of 500 to 1000 hp



and the gas leaving the Theisen disintegrator was clean enough for use in gas engine operation.

This Theisen scrubber was developed about a generation ago and, disregarding its heavy power requirement, has not been equalled by a scrubber of any other type up to very recently. However, within the last few years the Venturi scrubber followed by a Pease-Anthony scrubber has shown a capacity equal to the Theisen to collect fine particles, for example, soda fume from paper mill recovery furnaces, and fumes from reverberatory steel furnaces, with an overall efficiency of around 98 pct. The equipment, however, is about on a par with the Theisen as to power requirements and pressure drops—for example, on a paper mill, the combined Venturi-Pease-Anthony scrubber gas collecting sodium compounds, largely as fumes, and for a gas volume of some 45,000 cfm, required an induced-fan motor unit of between 225 and 250 hp.<sup>18</sup> The velocity of gases in the narrow section of the Venturi was about 300 fps and the pressure drop across the Venturi section something over 12 in. of water gauge. Its good efficiency is proportional to the power consumed, since the latter expresses the energy necessary to break up the water into independent small particles and it has been stated that, for good collection, the water particles should be reduced in size so that the diameter ratio between water particles and suspended dust or fume particle is not greater than 10 diam for the water to 1 diam of solid particle. It is evident if one is trying to collect particles of 1 micron and less a very fine subdivision of the wash waters is required and both the Theisen and the Venturi do that very thing but at a corresponding expense for power.

Packed towers (with coke or tile) have always given a good account of themselves but their efficiency is quite largely dependent on the pressure drop which again represents a power consumption to subdivide the water or increase its total surface, and the weakness of these packed towers is their tendency to choke with deposited solids.

The simplest design of scrubber, a coarse spray in a tower, is a good gas cooler but, except for the coarser particles of dust, is a poor collector. It uses relatively little power.

## FILTERS OR FILTRATION

Filters or filtration, as a means of dust and fume recovery, are very widely

used throughout industry, largely as baghouses. The temperature limit is quite low—for cotton bags it should not exceed 200°F and for woolen bags 250°F. Acid gases or corrosive compounds, of course, greatly shorten the life of bags but in very low concentrations or after partial neutralization, are encountered in many gases going to bag houses, for example, lead smelter gases.

A newer material for bag manufacture has become available during the last 10 years and gives promise, indeed, of great future service. This is glass fiber cloth, and while the earlier material tended to plug, new methods of weaving and new physical types of thread appear to offer fabrics having the filtering characteristics of wool, that is, filtering on the surface rather than through the pores of the medium. Present price and delivery are perhaps drawbacks but undoubtedly the future use of material such as woven glass fibers for baghouse service will become much more general, both for large and small units.

Bag house operation is generally on a basis of about 2 in. of draft loss but some are made to operate on 6 to 8 in. One difficulty is the passing of fume-like material through the bag weave at high pressure loss, while the same material may be satisfactorily collected at lower pressure drops, say 1 to 2 in. Cost of good baghouses or filter chambers are commonly less than electrical precipitators but still on the order of two to three times that of the best designs in centrifugal equipment. Filtration with a nearly new bag properly coated with a filter layer of particles removed from the gas stream and at a reasonable pressure drop is, nearly perfect, that is, nearly 100 pct. The efficiency varies downward from this as the bags get old and worn, or as pressure drop is increased and as rapping or shaking of bags must be done to prevent buildup of flow resistance across the bag wall. In bag house operation, where the bags are being shaken or rapped at regular intervals of time, there is usually to be noted a good "puff" to atmosphere as the rapping period starts. Some test work would indicate as very good practice a 95 pct collection efficiency, considered over a period of baghouse operation.

## ELECTRICAL PRECIPITATION

The last of the commercial types of equipment to be discussed is the *Electrical Precipitation* of suspended

particles in gases, perhaps as well known by the name of its inventor, the Cottrell process, after Dr. Frederick G. Cottrell, former professor of physical chemistry of the University of California, Berkeley.

You will observe from Miller's chart that its limit in degree of fineness of the material which it can collect far exceeds any other device or method of collection considered. Tobacco smoke, which is a tar fog, plus some carbon particles, is given a particle size of from 0.15 down to 0.01 micron, individually far too small to be seen under the highest magnification of a microscope. It has been stated that in the visible cloud exhaled from a cigarette there are some 5 million particles of tar fog per cc. In a laboratory apparatus the instantaneous precipitation of such tobacco smoke is quite a striking demonstration of electrical precipitation.

Strange as it may seem, the electrical phenomenon of precipitation as the result of a high potential direct current discharge and field, was a very early discovery. Hohlfield, in 1824, noted that a bottle filled with smoke and with a wire led into the bottle through the cork, could be almost instantly cleared when the knob of a charged Leyden jar was made to contact the wire into the bottle. That was at a date in history in which electricity was truly still a mystery and the only known way to generate it was the rubbing of a glass or wax rod with a silk or wool cloth.

As an industrial process it did not become of interest or successful until Cottrell, in the early years of this century,<sup>19</sup> applied our modern electromagnetic means of producing abundant high potential electricity. It is true a first effort was made at industrial application by Sir Oliver Lodge and associates in 1884-5, in attempting the precipitation of lead fumes in a lead smelter flue in Wales. His source of electrical power was a Wimshurst machine (developed in 1881) of two 5-ft glass plates driven by a 1 hp steam engine. We know now that such a Wimshurst machine could energize only a few inches of a pipe electrode and that to handle successfully the gases from an average lead smelter requires thousands of feet of electrode pipe and perhaps 50 to 100 kw of installed high potential electrical equipment for service therewith. The conversion of high potential alternating current readily obtained from modern power plant equipment into direct current (true, of an intermittent or pulsating type) by



means of a synchronous rotating make-and-break switch, was a major contribution by Cottrell and by this means he was enabled to pass to his electrically insulated high potential discharge electrode the necessary power to provide the electron or corona glow essential for dust charging and static precipitation.

While electrical precipitators for dust, fumes, mists and fogs have been built in a great variety of size, shape and outline designs, in essence they conform closely and are commonly either of plate or pipe design. The spacing between plates or the diameter of electrode pipes varies generally from 6 to 12 in., is tied directly to the available or desirable high voltage, and the latter, for general working conditions, is specified in the range of 50,000 to 100,000 volts. Always the high potential or discharging electrode possesses elements of small diameter, that is, from a No. 16 wire up to  $\frac{1}{4}$  in. rod from which there is obtained an electron discharge, commonly visible as a corona glow. Such electron emission produces a rupture stress upon the electrical neutral gas molecules immediately adjacent to the discharge electrode, and while the positive charge must fall into the negative discharge electrode, the negative charge, i.e., the electron charge attaches itself to a gas molecule and such charged gas particle under the strong electrical field between the discharge and collecting electrodes starts to migrate to the collecting electrode. As it encounters dust or fume particles it either gives up its charge thereto or the charged gas particle is adsorbed or attached to the dust particle and thereby the suspended dust or fume particle is moved toward the positive or collecting electrode. Upon reaching the collecting electrode such charged dust particle should give up its charge and become grounded and form one particle of a cake which may later be rapped or shaken down into a hopper beneath the plate electrode. The readiness of suspended particles to receive a charge, to release such charge upon reaching the electrode of opposite polarity and the mechanical adherence of deposited particles, all are modified and are increased or decreased by conditions within the gas stream. Therefore for "poorly conducting dusts" it is common to "condition" the gases and particles, that is, improve their collectability by adding to the gas stream such agents as moisture, acids, bases, salts, oils, and others. In general

each installation and the operations to which it is attached provide a special problem and require specific handling.

With the provisions as outlined above, the field of collection of industrial dusts and fumes is broad indeed and the actual application and plant installation are ever widening and the units in service always increasing in numbers.

Since its cost of installation is several times that of cyclonic equipment or a settling chamber, its use is justified only where the amount and value of recovered products are large or where the degree of gas cleaning is of the highest importance. Good examples of the latter are to be found in the gas cleaning necessary for contact acid manufacture from gas produced in the roasting of mineral sulphides, and the cleaning of iron blast furnace gas for use as a dust and fume-free fuel in stoves and coke oven retorts.

One phase of electrical precipitation not covered in the preceding discussion, is its use in the purification of air for human habitations. Here the recovery in the precipitator may be bacteria, pollen and the like, as well as minute floating particles of solids and mists. Its construction and operation vary from those of the usual commercial plant by the use of very much reduced spacing and corresponding voltage reduction and the use of very fine wires for ionization and of a double plate field for the static collection of the charged particles. It is essential in this field that ozone and oxides of nitrogen be reduced below detectable amounts, which is accomplished by the relative low voltage and the use of a static field following upon a gas and dust-charging field.

### **V—Description of Equipment of the Several Types Discussed Previously for Dust and Fume Collection**

#### **SETTLING CHAMBERS**

In 1902 the Anaconda Copper Mining Co. completed a new copper smelter which, at that time, was the largest and the best equipped of any such type of plant in the world. In design and provision for the possibilities of the future, no plant built had had better engineering or more skill of design. Each unit of operation such as roaster, blast furnace, and converter department had its own stack and its own dust collection settling chamber. These

chambers, approximately 40 ft wide, 40 ft high and 260 ft long had a net height for dust settling of 30 ft, since about 10 ft from ground level a chamber floor was installed and below this was a system of tracks and means of dust removal and transport. Since the furnace feed, as compared with today's, was relatively coarse, comprising crude ore and the output of the best of gravity mills of the day, the settling chambers gave a very good account of themselves on the basis of copper recovery, but still there was a stack discharge which reached the valley areas well below the smelter level and several miles away, which brought on complaints of harm to livestock and growing crops. To secure still more provision for settlement, for cooling and condensing of certain constituents and to discharge the smelter gases at a greatly increased height above the valley, a combined settling chamber and flue for the combined smelter gases was carried up a hillside, the lower section being 60 ft wide, 20 ft on its side walls, both sides sloping to the middle with an underground cleanout tunnel on the middle line with 36 ft from top of flue to top of tunnel. This lower section was built approximately 1000 ft long and with a brick jack arch roof. The upper 1000 ft was made just twice as wide and twice the cross-section and with a steel top to promote condensation, particularly of the arsenic trioxide, the constituent of the stack gases to which most objection had been raised. This entire system of settling chambers and extended flues comprised about 6,000,000 cf of settling space and represented a heavy investment even for those days. Due to the acid character of the gases, the only other competing equipment of that date, the baghouse, was entirely out of consideration, and the system installed did represent the best of engineering and scientific application.

Nevertheless the complaints continued and the evidence of operation indicated the inability of a settling chamber to collect satisfactorily the "fumes" or volatilized and condensed metallic compounds such as the oxides of arsenic, lead and antimony. Years later, to bring the collection of all products up to a high degree of recovery, an extended Cottrell or electrical precipitation installation was built to operate in series with and after the settling chamber system. Its collection, largely fumes, brought the overall recovery of both dust and fume from



furnace to stack to the present highly efficient operation.

An interesting modification or improvement of the simple settling chamber is the wire-hung dust chamber of which the largest was that installed at the Boston and Montana Company's Smelter plant at Great Falls, Montana, in 1908 or 1909.<sup>20</sup> As a matter of fact a wire-hung dust chamber is really a combination of a settling and an inertial type of recovery equipment, since the gas stream in striking the wire must sharply turn while the dust particles move forward and strike the wire surface, yielding their energy thereto and falling. This wire-hung dust chamber was 177 ft wide, 367 ft long, 21 ft high. It had a completely hoppers bottom (over 1000 hoppers) with wires attached to and guided by heavy screens of about 2 in. mesh. Over one million wires in two banks were hung in this chamber. A mechanical rapping device was provided for the suspended system of vertical wires. Its collection efficiency, on total solids, was about  $3\frac{1}{2}$  times that of the simple open dust chamber and it undoubtedly collected more efficiently on the finer fractions; but in course of time, due partly to shutdown periods and consequent corrosion, it was found too expensive to maintain the wire hung feature of construction and the wires were removed. This settling chamber, without its wires, operated for a number of years but eventually was replaced by a Cottrell, the latter making use of hoppers and supporting steel work of a portion of the old chamber.

#### INERTIAL OR CENTRIFUGAL

For the inertial or centrifugal type of collectors it is proposed to discuss briefly three installations, two on boiler plant gases and one on converter dust recovery.

Modern combustion of coal in a power house required the coal to be finely ground and then burned in suspension. This results in most of the ash appearing in suspension in the gases as finely divided small spheres, some hollow, and of which a fair proportion—say a third—may be in the size range of 1 to 10 microns. The percentage of the ash leaving the boiler as so-called "fly ash" is usually better than 80 pct of the total ash and occasionally some plants get into the 90 pct class of ash to stack gases. While the huge central station plants with their requirement of overload for limited periods have generally, in the past, used electrical precipitators, some of

the new plants today are choosing a combination of cyclonic tubes followed by an electrical precipitator. This mechanical collector between the boiler and the precipitator, for a 1,000,000 lb per hr boiler with over 400,000 cfm of flue gas, comprises a series of Multiclone units all operating in parallel from a common flue of boiler width, there being 5 units of Multiclones, each unit with 156 tubes in parallel position, the tubes being of a uniform inside diameter of 9 in. and each tube provided with a stationary cyclonic vane at its top and inlet heading.

Plants of an industrial nature are not usually so severely restricted as to amount of emission and an example without the auxiliary electrical precipitator, in series, is a sugar mill at Vancouver, B.C. It is an excellent example of a reasonable efficiency utilizing a small-diameter type of cyclonic collector with a multiplicity of tubes. The gases derived from three boilers at this Vancouver plant and totaling approximately 70,000 to 80,000 cfm, pass to a battery of Multiclone units, each unit of 30 tubes and each tube 9 in. in diam. A system of dampers is provided for the Multiclone units so that as a boiler is taken off or put on the line the number of Multiclone units in service can be proportionately varied. Normally the pressure drop across the Multiclone is maintained at 2 to 3 in. of water column. The pulverized coal is of local supply of relatively high ash and usual efficiency is on the order of 85 pct.

Multiclones with a larger diameter of tube, that is, 16 in. diam tubes have found service at several copper smelter converter installations in the West. As an example, there may be taken the installations at the McGill, Nevada, Smelter of the Kennecott Copper Corp. Here 4 units of Multiclones, each of 45 tubes 16 in. in diam are installed to serve three converters. The gas volume approximates 273,000 cfm at 600°F. For movement of the gases through the flue system and to supply the approximately 3 in. pressure drop across the Multiclones, a large induced draft fan is located on the exit flue from the Multiclone units and is driven by a 150 hp motor. Due to the corrosive nature of converter gases at lowered temperature, ample heat insulation has been provided, mainly in the form of a rock wool blanket 4 in. in thickness. On the copper content only of the converter dust and fine discharge, the Multiclone collects 90 pct or better but on the overall of dust and fume the

collection is considerably below the copper collection.

#### FILTRATION

The third method of dust collection to be illustrated by plant example is that of baghouse and we have selected two baghouse installations; one at a copper smelter and one at a lead plant.

While present-day practice does not favor a baghouse on copper plant gases but does favor its use at lead reduction works, it is of interest to note an example of a baghouse installation on a copper plant works which for its day and time was a signal success. Early in this century the Mammoth Smelting Co. at Kennett, California, operated a copper smelter of 4 blast furnaces and 2 converters. The copper ore, a sulphide, also carried enough zinc so that the gases were approximately free of  $\text{SO}_3$  or  $\text{H}_2\text{SO}_4$  and, by lowering the temperature by radiation-cooler pipes, plus further cooling by dilution with outside air, successful operation on the baghouse was obtained. The installation of the baghouse was not necessitated by plant requirements as the collected product was not returned to furnaces, and unless it has been washed away or dissolved by the rains since the plant was shut down about 30 years ago, it is still on hand at the smelter site. Its construction was due to a desire of the smelter company to meet the objections to smelter dust fall from communities in the upper end of the Sacramento Valley.

The gases from the furnaces averaged about 600°F and were passed to a radiation cooler consisting of a number of 2-ft pipes operating in parallel, which discharged to the inlet flue of the baghouse. Into this same header flue a fan forced outside air and the resulting air-smelter gas mixture was maintained at about 200°F. The baghouse discharged to atmosphere through the 5 large square stacks. Approximately 3000 woolen bags, 18 in. diam by 35 ft high were installed in the baghouse and the gas volume, as cooled and diluted to approximately 200°F, amounted to 350,000 cfm. The tonnage of ore smelted amounted to approximately 1000 tons per day, and the baghouse collection to 18 to 20 tons per day.

Typical baghouses for lead smelter service have been described by several authors and the one in service at Murray Smelter<sup>21</sup> in the Salt Lake area is representative of good design and good construction. The housing for the bag sections is of heavy brick construc-

tion and has outside dimensions of 216.5 ft long, 90.5 ft wide and a height to roof trusses of 51.5 ft. The lower 16 ft of wall is 21 in. thick, the next 18 ft 17 in. thick and the remaining height 13 in. wall. Forty-eight brick buttresses are spaced along the walls and add stiffness and rigidity to construction. There are four bag compartments separated by brick partition walls from floor line to roof trusses. Each compartment below the chamber floor is separated into 4 divisions by brick walls, each with its own cleanout door. Installed in the four compartments is a total of 4032 bags, each bag 18 in. diam and 30 ft long and these bags are made of wool to very careful specifications. As originally built there was provided 570,012 sq ft of filtering area to handle 165,000 cfm of gas or at the rate of 3.45 sq ft of bag surface to 1 cf of gas per min. Mechanical rapping or shaking of the bags was provided at their top ends.

Some later and smaller baghouses at lead smelters have been able to increase greatly the ratio of cfm per square foot of area by means of fully automatic operation of damper and bag shaking, some reporting a ratio of 1 to 1. In the case of a true fume such as arsenic or lead, however, increasing velocity through the filter cloth openings tends to cause increased escape of unagglomerated particles, and so increased economy of investment is offset by somewhat lower efficiencies. However, it is to be recollected that baghouses at lead smelters are as much a consideration for employee and community health as they are an investment in equipment to provide an economic return, and therefore are always planned for relatively ample capacity and high efficiency.

## SCRUBBERS

The fourth means of collection, that is, scrubbers, "disintegrators," and the like, making use of water as a medium to collect fume and dusts, have rarely found practical use at lead and copper works except for the small volumes encountered in silver and gold refining, and this, no doubt, has been largely due to the corrosion hazard. More than half a century ago, around iron blast furnaces, they received much attention and many units were installed. The combined inertial and wetting procedures used, for example, the Theisen disintegrator gas washer, were the most efficient collectors on the market and

collected not only the dust, but also the zinc and alkali fumes which often appear in the iron blast furnace gases. In principle, this Theisen scrubber is a fan with the usually solid blades replaced by perforated plates or a heavy screen mesh. Water is introduced with the inlet gases near the center of the fan runner and is broken up into fine particles in passing through or from center to periphery of runner. To collect particles of dust and fume, the water particles must be on the order of not over 10 times the diameter of the dust or fume particles and where particles of around 1 micron or less are to be collected, an enormous subdivision of water particles is required and this subdivision requires extraordinary high power consumption. In the older steel works of continental Europe, often the largest power unit of the works was attached to the Theisen washer and from 2 to 10 hp per 1000 cf of gas treated was the rule. The Theisen disintegrator, however, did prepare iron blast furnace gas for combustion in gas engines with a content in solid suspended particles not far from that of outside air. Today their use has greatly decreased, the same degree of cleaning being secured by a film type of electrical precipitator with a power consumption only a few percent of that of the Theisen. Nevertheless, it should be noted that at the time of its first introduction, 50 years or more ago, it was an outstanding success and for the service it rendered over a generation or more, no other equipment of that period was in its class.

However, within the last few years a new scrubber has been brought on the market, known as the Venturi<sup>17</sup> which, combined with the Pease-Anthony scrubber<sup>16</sup> shows reported efficiencies on the order of 98 pct on paper mill fume. On reverberatory open hearth waste gases containing fumes and the like, equally good efficiencies have been reported. In the Venturi scrubber a high velocity of gas stream is maintained at the throat, about 250 to 300 fps with a corresponding pressure drop across the throat section approximating 12 in. of water column. A series of water connections surround the throat of the Venturi, and in the throat conditions are such that an extraordinarily violent turbulence is set up, sufficient to provide for the wetting and collection into the moving water gas mixture, of the dust and fume particles suspended in the gas stream. From the Venturi the gases

either pass to a simple cyclone for separation of water particles and gas, or to a Pease-Anthony scrubber, which is a cyclone with tangential gas entrance near the bottom and a central column with an array of spray heads discharging in such a manner that the longest possible path in the cylindrical shell is obtained for spray discharge and passing the gas stream. On paper mill (soda) fume, the Venturi alone has been reported as collecting approximately 90 pct and the combined Venturi and Pease-Anthony 98 pct of the suspended fumes in the gases. The latter figure and also that for the combined Venturi-Pease Anthony have been reported on an open hearth reverberatory with oxygen in the bath, the latter to speed the refining operation at top temperatures. The drawback of the Venturi scrubber, like that of the Theisen, is the high power consumption required for induced fan and to provide the throat velocity or the pressure drop across the Venturi and the Pease-Anthony. About 220 hp was used on the reverberatory tests handling approximately 45,000 cfm. Should it prove practical to reduce the power consumption without decreasing the efficiency appreciably, this type of scrubber should have most excellent industrial possibilities.

Excepting these two types of Theisen and Venturi which do collect fume, the general run of scrubbers is used on dust particles (say, 100 to 1000 times the weight of a fume particle) and there they do acceptable work with relative low power consumption. Probably best in practice are the countercurrent tile-packed towers, although commonly these require periods of special flushing or cleaning to prevent choking and buildup. They are generally planned for a 1 to 3 in. pressure drop across the tile sections but with only a small buildup of mud or caked material will promptly double or triple their pressure drop.

On hot gases and series operation of two or more spray chambers, the first at a higher temperature, the second cooling the gases below dew point, it is practical to vaporize more moisture into the first chamber than the gases can retain in the cooler conditions of the second spray chamber with the result of the condensing out of a proportion of the water vapor produced in the first chamber. Such condensed precipitates make use of fine particles of dust and fume as nuclei for their condensation and thus permit of high effi-



ciencies. On a calcining kiln, where only a fine dust escaped, a reported 97 pct removal of suspended solids in gases was reported in this manner of operation.

The great objection to scrubbing in general is the need to care for large volumes of water containing a relatively small amount of solids. Thickeners, filters, storage tanks and extensive pumping and piping are commonly required to complete a properly operating plant.

### ELECTRICAL PRECIPITATION

We now come to the fifth and last method under discussion for dust and fume collection, *viz*, *Electrical Precipitation*. So many plants have been described in the literature that it seems questionable if anything new may be offered here. However, we propose to describe briefly two types of precipitators: (1) that aimed at collection of large quantities of dust or dust and fume, and (2) that aimed at the final cleanup of a gas so that such cleaned gas may be advantageously used in further processes or equipment.

For the first classification above, it is proposed to describe an electrical precipitator recently installed at a cement plant in the South, and a power plant precipitator going into service in the near future. For the second classification above or purification of gases it is proposed to describe a plant installed to "fine"-clean iron blast furnace gas and a plant to clean roaster gas for contact acid manufacture.

The cement plant of the Universal Atlas Cement Co. at Leeds,<sup>22</sup> Alabama, is not far from the center of the town and while producing only at a minimum or average capacity did not discharge from its stack an objectionable quantity of dust. Along with the war and the aftermath of war, with its huge demand for cement, came the necessity to increase to the limit the plant's capacity to produce and, with this increase, came immediate evidence of greatly increased dust discharge from the stacks. To meet the desired reduction in stack emission of solids a three-unit precipitator was installed, each unit to handle 100,000 cfm at the prevailing temperature of approximately 700°F. The actual efficiency for slightly less than the rated gas volume was 94 pct or better. Each of the three units of precipitator are provided with three sections or electrode banks in series, the first two sections being of 15 gas passageways or ducts on 8 in.

centers and the last section of a V or pocket type with 12 gas passageways or ducts on 10 in. centers. Each collecting electrode approximates a dimension of 8 by 18 ft, the discharge electrodes being square twisted rods  $\frac{1}{8}$  in. in diam in a supporting frame. Six special electrical units each with a 75,000 volt transformer and mechanical rectifier serve the installation. Automatic or push button controls are provided for all operating equipment including rapping both discharge and collecting electrodes, dampers, screw conveying equipment and the like. The return of the dry dust to a wet process plant is always a difficult problem and here 55 pct of the collected dust is returned as a dry feed to the center of a vortex of slurry just as it enters the feed pipe to kiln.

Power plants and boiler sizes are ever growing larger and in some of the newer power plants gas volumes exceed anything but those of our very largest copper smelters. The boilers of huge size now being installed are possible because of powdered fuel combustion developments and since, in powdered fuel plants, the major portion of the ash is suspended in the spent gases from boiler and normally passing to stack, collection equipment for ash particles has become an essential requirement. Electrical precipitation is generally favored because of the degree of fineness, (20 to 50 pct minus 10 microns) of the ash and the fact that its collection is dry, thus avoiding a corrosion problem.

At the present time there is under construction a power plant involving three boilers each of one million lb steam evaporation per hour and each producing somewhat under  $\frac{1}{2}$  million cf of gas as measured at exit of air heater at approximately 300°F. Each boiler gas discharge has its own collection installation and a brief description follows: The collector is a combined inertial and electrical precipitator in which the electrical precipitator for each boiler is preceded by a Multiclone installation as described under the heading of inertial or centrifugal equipment. The precipitator for each boiler is divided into five units, each with an outlet damper and each precipitator unit consists of 15 gas passageways or ducts, and while only one section long, each duct carries three collecting plates, each plate 3 ft wide by 16 ft high with 2 ft of settling and agglomerating space between each two plates in the direction of gas flow. The duct width,

or from center to center of two collecting plates, is 9 in. Discharge electrodes consist of the usual square twisted  $\frac{1}{8}$  in. bars. The gas volume per boiler is 400,000 cfm at prevailing temperature of 325°F and the total for three boilers is 1,200,000 cfm at 325°F. For the three-boiler plant there is a total of 2340 Multiclone tubes preceding the precipitator and a total of 225 gas passageways or ducts in the electrical precipitator. This gas volume is larger than the usual nonferrous metallurgical plant except for several of the very largest copper smelters. Eventually the power plant will have a fourth and a fifth boiler, each of one million lb per hour production of steam or perhaps even larger—a million and a quarter is even under discussion.

From collection of the major portion of a nuisance waste at a power plant with a huge gas volume, we turn now to a plant handling a much more limited gas volume, a mere 100,000 cfm, but which is required to clean the gas almost to the equal of outside air, that is, to 0.007 grain per cf or better. This is entirely an operation of purification of the gases and the recovered material, mostly fume, is of no value or use.

Modern steel works find it profitable to have the cleanest obtainable fuel gas from iron blast furnace operation in order (1) to prevent slagging of the checker work in the stoves, (2) to permit closer packing of refractory brick in stoves, (3) to permit satisfactory operation of automatic air and gas regulators on blast furnace stoves and of fuel at coke oven plants without danger of slagging the brick work. All this they obtain with a train of collection equipment on the gases coming from the iron blast furnace top. The train consists of (1) a dry dust catcher or chamber where the very coarse dust is caught and where dust concentration is reduced from around 12 grains per cf to around 3 or 4 grains per cf. (2) The gases then enter a scrubber provided either with a checker work of wooden strips or a series of sections of tile packing with heavy coarse sprays at top and commonly at two or three intermediate points with gases passing upward and water downward, that is, countercurrent operation. The gas entering the scrubber has a concentration of 3 to 4 grains and the gas leaving the scrubber has a concentration in solid suspension of 0.25 to 0.3 grain. From the scrubber the gases enter the precipitator and are cleaned in the

precipitator down to a concentration of approximately 0.007 grain per cf. This reduction in suspended solids of 0.3 to 0.007 corresponds to an efficiency of 97.7 pct. From the precipitator the clean gases go to the receiver and to special services where extreme cleanliness is desired. The gas volume at precipitator approximates 100,000 cfm and 4 units of precipitator are placed in two circular shells, each with a division wall down its center. Each unit has 126 pipe electrodes 8 in. in diam by 15 ft long or a total for the 4 units of 504 tubes. Three electrical sets, each 25 kva, with 75,000 volt transformers supply the special direct current required for precipitator operation.

A second field where electrical precipitators are used in the purification of gases is in the manufacture of sulphuric acid by the contact process with the  $\text{SO}_2$  derived by combustion of the usual run of sulphide ores or sulphide concentrates—commonly zinc, iron, or copper sulphides or mixtures usually with minor amounts of arsenic, antimony, lead and the like.

Arsenic oxide, and some of the other constituents, poison the platinum catalyst and others merely coat over the active mass, so it is necessary to have the gas, before going to the  $\text{SO}_2$  to  $\text{SO}_3$  converters, cleaned down to a few parts in 100,000 of arsenic figured as  $\text{As}_2\text{O}_3$  and as grains per cf. This is accomplished by a thorough wetting and cooling of the roaster gas in towers and scrubbers of various design, occasionally a packed tower is required, and the passage of the gas at 80 to 100°F to a lead type precipitator. The gases reaching the precipitator are fully saturated with water vapor and contain in addition mist particles of very weak acid. These latter usually contain the minute particles of arsenic probably as the nuclei of mist particle formation. In one acid plant with a gas volume of about 7500 cfm at the prevailing temperature of 80 to 100°F, a precipitator was used consisting of 72 pipes, 70 of these 10 in. in diam by 12 ft long and two pipes carrying stiff rods to lower grid, 13 in. diam by 12½ ft high.

The actual precipitate was a very light liquid sludge which drips from pipes and eventually finds its way to a waste sump. One electrical set with a 75,000 volt transformer and two mechanical rectifiers (one a spare) serves the pipe section. The discharge electrodes are lead-covered iron wires in a star cross-sectional design and held straight and true by lead weights. Many lead pipe units of the same general design are used at acid-concentrating plants such as are to be found at munitions works and occasionally at chemical and oil works. Both material savings and improvement in health conditions are derived benefits from the precipitator operation on waste acid gases.

## Conclusion

It may be true we have tried to cover too much, too sketchily in this paper. However, it is thought the object in view, viz, to acquaint our member engineers both with a birds-eye view of the problems of collection of suspended dusts and fumes as a whole and also to offer enough of the practical in description of types of equipment and specific installation as will permit of fair engineering visualization, has been reasonably well accomplished. It is recognized that books have been written and more are in prospect on particular phases of the subject matter here handled and that is as it should be, for truly to meet the modern requirements in dust and fume control in mines and mills, in industrial plants and smelters, will require some technical knowledge of the problem by nearly all engineers and a great deal on the part of those directly involved in collection. Also this latter class in industry will constitute a staff many times that of today and the expenditure for equipment will approximate hundreds of millions and this is limited to the near continental area of our United States.

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# Some Observations on the Recovery of Cold Worked Aluminum

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## Introduction

The phenomenon of recovery of cold-worked metals is interesting not only because of its practical importance but also because of its fundamental significance in solid state reactions. Although extensive investigations<sup>1,2</sup> have already been made in an attempt to discover the mechanics of the recovery process, many of the observations have not yet been satisfactorily correlated to provide a completely consistent model for the process. The wide differences in the recovery rates of various properties can be cited as a typical example of one of the difficulties that are encountered. Frequently, for example, the electrical resistance will have almost completely recovered before any recovery in tensile strength can be detected. Of course, such differences in the recovery rates of different properties might be explained by assuming that each property is a unique function of the work-hardened state, and consequently each property exhibits its own unique recovery rate. The assumption that different properties are uniquely related to the work-hardened state cannot be denied. On the other hand, the properties that recover at different rates often exhibit more or less parallel changes upon work-hardening. This suggests that the microstructural changes attending recovery are not exactly the reverse of the changes attending work-hardening. Several types of imperfections must be postulated in order to account for this apparent anomaly. The different recovery rates for various properties, then, are due to the different recovery rates of the type of imperfection to which each property is most sensitive as well as the unique dependence of each property on the cold-

worked state. This concept assumes that a simple model of the work-hardened state consisting only of one type of imperfection, such as Taylor's type of dislocation patterns, is inadequate to cope with the diversified phenomena attending work-hardening and recovery.

Although current models for the work-hardened state are not useful for describing all aspects of the recovery process, the general trends of the recovery of each postulated type of imperfection as a function of time and temperature should be at least qualitatively deducible from the rather well developed laws of kinetics of reactions in the solid state. Consequently, recovery data might prove useful for elucidating some aspects of the complexities of the work-hardened state of metals. A preliminary attempt to study work-hardening by investigating recovery rates of cold-worked metals is outlined in the following pages of this report.

## Experimental Procedure

Many properties recover when cold-worked metals are annealed below their recrystallization temperature. Therefore, electrical resistivity, thermal electromotive force, X ray diffraction line

widths, X ray diffraction line intensities, elastic spring back, density and other physical and chemical properties have been used to study the recovery process. Major interest, however, has generally been directed toward the recovery of the mechanical properties such as hardness, yield strength, and tensile strength. But a search of the literature suggests that the effect of recovery on the true stress-true strain curve has been neglected, in spite of the current recognition of the fundamental importance of such an investigation. An investigation on the effect of recovery treatment on the true stress-true strain curves in tension, therefore, was undertaken in the present study.

Commercially pure aluminum (99. + pct Al) in the form of 0.100 in. thick rolled sheet of 2S-O aluminum alloy was selected as the material for this investigation because rather extensive correlatable data are already available on the recovery of some of its properties. Tensile specimens having a 6 in. long gauge section and a uniform reduced section width of 0.500 in. were machined from the sheet in accordance with a design that has previously been reported.<sup>3</sup> All specimens were selected with their axes aligned in the rolling direction. In order to eliminate the effects of previous work-hardening and the effects of machining, the specimens were annealed for 15 min. at 750°F before testing.

During tensile testing the loads were measured by means of a proving ring (sensitive to ½ lb) in series with the specimen.<sup>4</sup> Strains were determined from the extension of a rack and pinion strain gauge sensitive to a strain of  $\pm 0.0001$ . The stress was recorded as the true stress, namely

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TP 2721 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before April 1, 1950. Discussion is tentatively scheduled for publication in November 1950.

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<sup>1</sup> References are at the end of the paper.

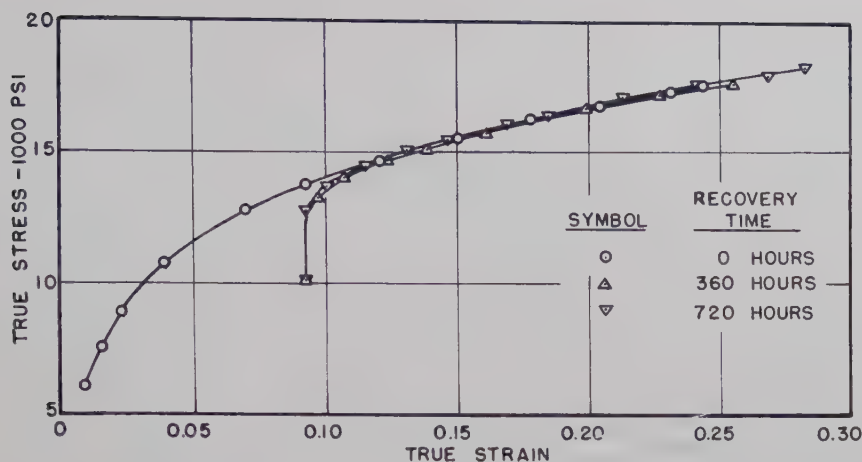


FIG 1—Recovery of 2S-O alloy at 90°F after a prestrain of 0.092.

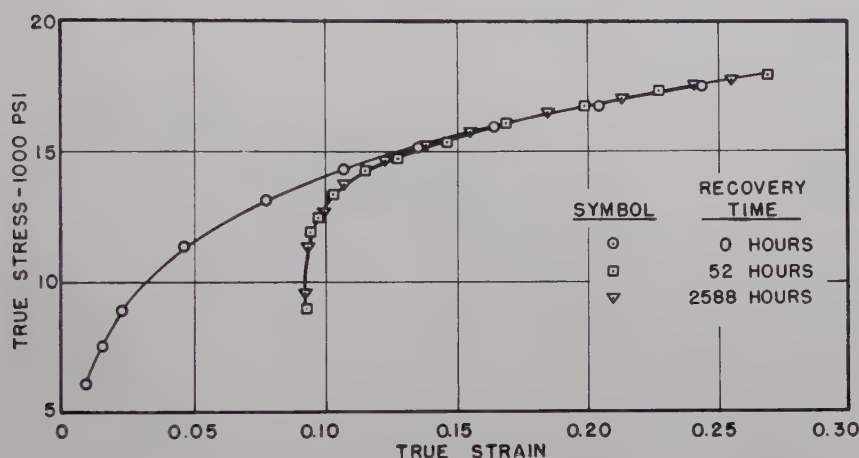


FIG 2—Recovery of 2S-O alloy at 212°F after a prestrain of 0.092.

$$\text{Stress} = \frac{\text{Load}}{\text{Instantaneous Area}}$$

and the total true strain was evaluated by

$$\text{Strain} = \log_e \frac{\text{Instantaneous Length}}{\text{Initial Length}}$$

The true plastic strain was then calculated by subtracting the known elastic component from the total true strain.

## Experimental Results

Several typical experimental results are given in Fig 1 to 4 in the form of true stress-true plastic strain curves. The upper curve of each figure is the work-hardening curve for the annealed specimens. The remaining curves were obtained from specimens that had been strained to  $\epsilon = 0.092$  at atmospheric temperature, recovered for the recorded time and temperature, and then retested at atmospheric temperature. Only a few representative data are shown in Fig 1 to 4. Other prestrains

(0.048 and 0.125) and intermediate times of annealing yielded analogous results.

## Discussion of Results

The data recorded in Fig 1 reveal that cold-worked aluminum recovers upon annealing at 90°F. The recovery rate is quite rapid initially, but it ceases before complete recovery of the initial flow stress is achieved. It is significant to note, however, that upon restraining only the initial flow stress appears to have recovered, and after an additional strain of about 0.04 the stress-strain curve of the recovered material coincides with that of the virgin annealed specimen at the same total strain. Consequently those imperfections that are readily activated at this low recovery temperature are readily reintroduced upon restraining. The exact coincidence of the latter portion of the stress-strain curve of recovered specimens with the curve for the virgin

annealed material at the same total strain suggests that the work-hardened states in the two cases are substantially identical.

As shown in Fig 2 the phenomenon of recovery at 212°F is qualitatively analogous to that which occurs at 90°F. The only significant difference is that a greater amount of recovery of the initial flow stress results at the higher annealing temperature. Nevertheless the constant value of the initial flow stress is yet above that for complete recovery.

The term metarecovery will be used to describe the type of recovery experienced by cold-worked aluminum 2S-O alloy upon annealing at 90 and 212°F. Metarecovery, then, consists of the removal of readily activated imperfections at low recovery temperatures. The removal of these imperfections causes an appreciable decrease in the initial flow stress; they are again readily restored upon restraining. After small additional strains (about 0.04) have been imposed on the recovered



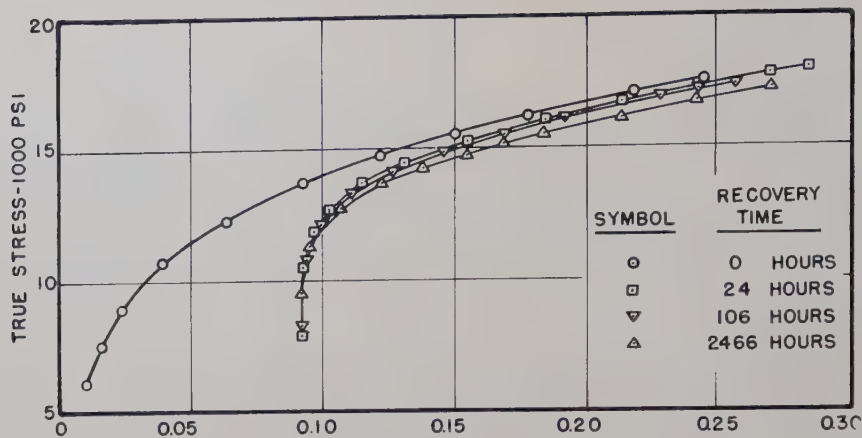


FIG 3—Recovery of 2S-O alloy at 300°F after a prestrain of 0.092.

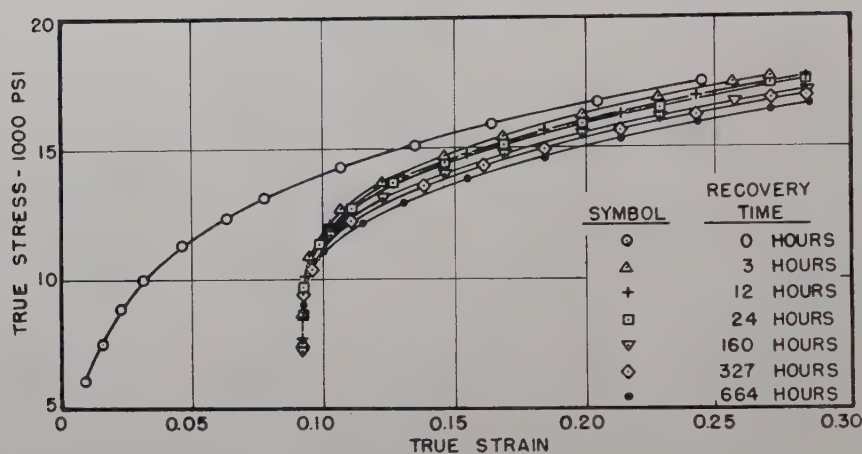


FIG 4—Recovery of 2S-O alloy at 400°F after a prestrain of 0.092.

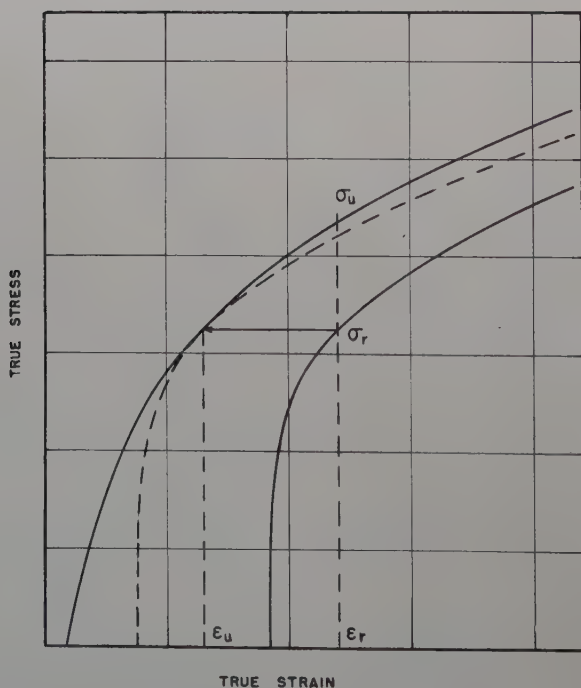


FIG 5—A method of evaluating the amount of orthorecovery.

specimen it responds plastically in a manner identical with that of a virgin specimen which has been deformed to the same total strain. Metarecovery takes place at a high initial rate but the specimen soon acquires constant plastic properties before complete recovery occurs. The amount of metarecovery, as dictated by the reduction in the initial flow stress below the value in the work-hardened specimen, is greater the higher the annealing temperature. Two possible interpretations can be ascribed to this observation. If the type of imperfections that are removed upon metarecovery do not interact with each other, they must individually have activation energies for recovery over a broad band of values. The imperfections having the lower values of activation energies are then removed at the lower annealing temperatures, whereas higher annealing temperatures are required to remove those imperfections that have higher activation energies. In this way it is

possible to account for the early termination of metarecovery before complete recovery is achieved. On the other hand, the imperfections might exhibit interactions such that the activation energy for metarecovery is a function of the density of the imperfections. In order to account for the observations it must be assumed that the activation energy for recovery increases rapidly with a decrease in the density of imperfections. The present evidence does not permit identification of which of the two alternate mechanisms of metarecovery is correct; it is interesting to note, however, that only the second mechanism is compatible with recovery of Taylor's stable patterns of edge type dislocations.<sup>5-10</sup>

The stress-strain curves given in Fig 3 and 4, for specimens annealed at 300 and 400°F, appear to be slightly different from those shown in Fig 1 and 2. They not only exhibit the previously described metarecovery, as indicated by the decrease in the initial flow stress after annealing, but they also show permanent effects of the annealing treatment. The flow stress after recovery at these higher temperatures never acquires the same value as the flow stress of the virgin material at the same total strain. Beyond an additional strain of about 0.05 after recovery treatment at these higher temperatures, the stress-strain curves for the recovered specimens fall below but parallel to that for a virgin annealed specimen at the same total strain. It will be convenient to describe this type of recovery as orthorecovery. Two important characteristics serve to distinguish orthorecovery from metarecovery. First the true stress-true strain curve of a material which has been subject to orthorecovery is below that for the virgin material at the same total plastic strain for all strains. And secondly, whereas orthorecovery appears to continue until complete restoration of the annealed properties is achieved, metarecovery practically ceases far short of complete recovery. This conclusion is immediately apparent when it is noted that even after annealing 2466 hr at 300°F or after 664 hr at 400°F additional orthorecovery is yet occurring.

It is not immediately apparent as to how a quantitative measure of orthorecovery might be obtained. It might be thought that shifting the stress-strain curve of a partially recovered specimen to the left would bring the latter portion of the stress-strain curve

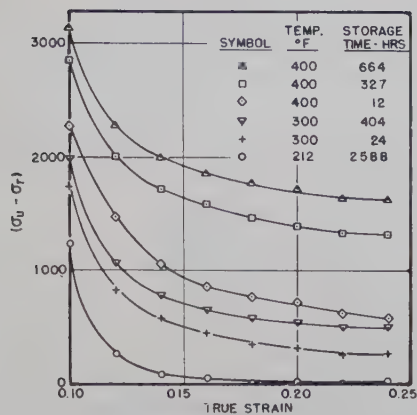


FIG 6—The reduction in flow stress upon recovery as a function of total strain.

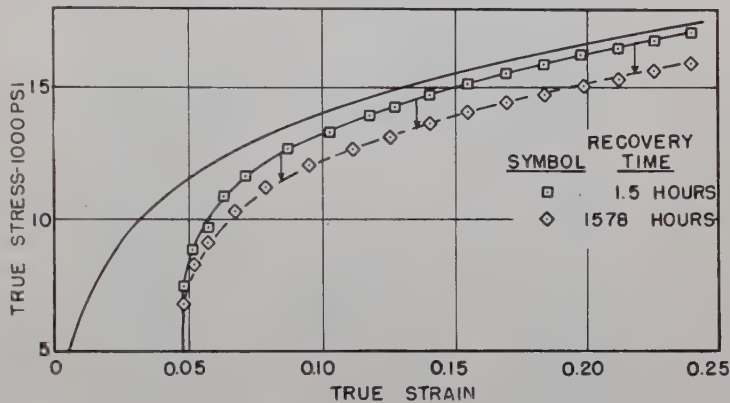


FIG 7—The effect of recovery at 400°F on the stress-strain curve for 2S-O alloy prestrained 0.048.

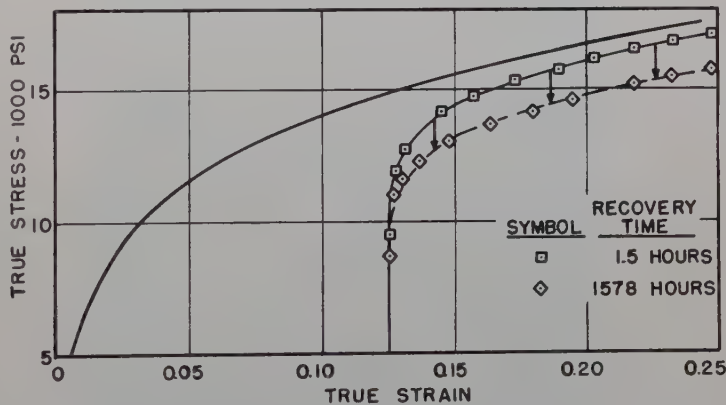


FIG 8—The effect of recovery at 400°F on the stress-strain curve for 2S-O alloy prestrained 0.125.

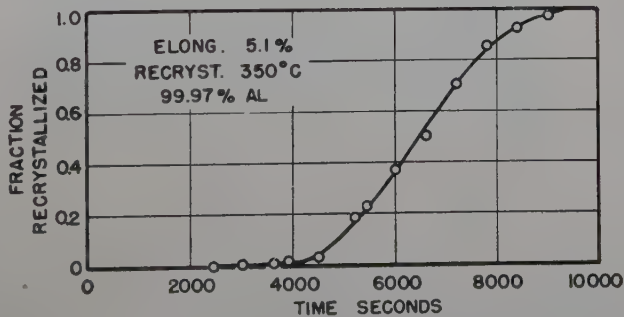


FIG 9—Typical curve for fraction recrystallized vs. time relationship. (After W. A. Anderson and R. F. Mehl.)



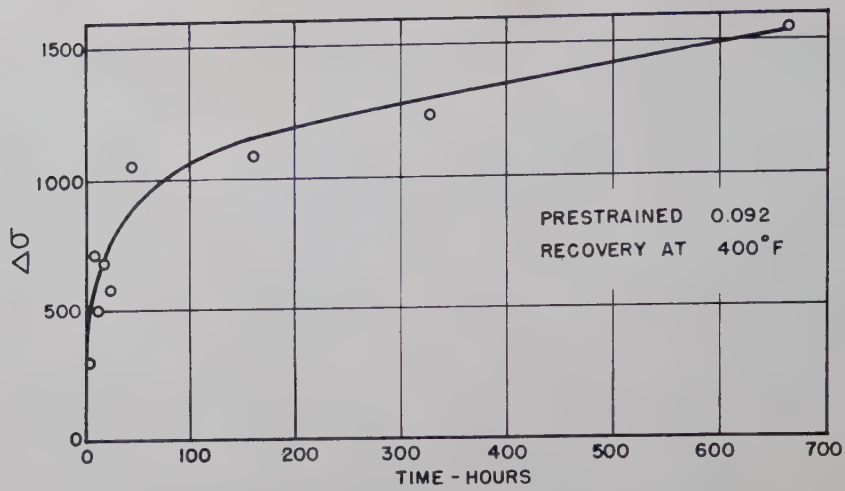


FIG 10—The effect of annealing time on orthorecovery.

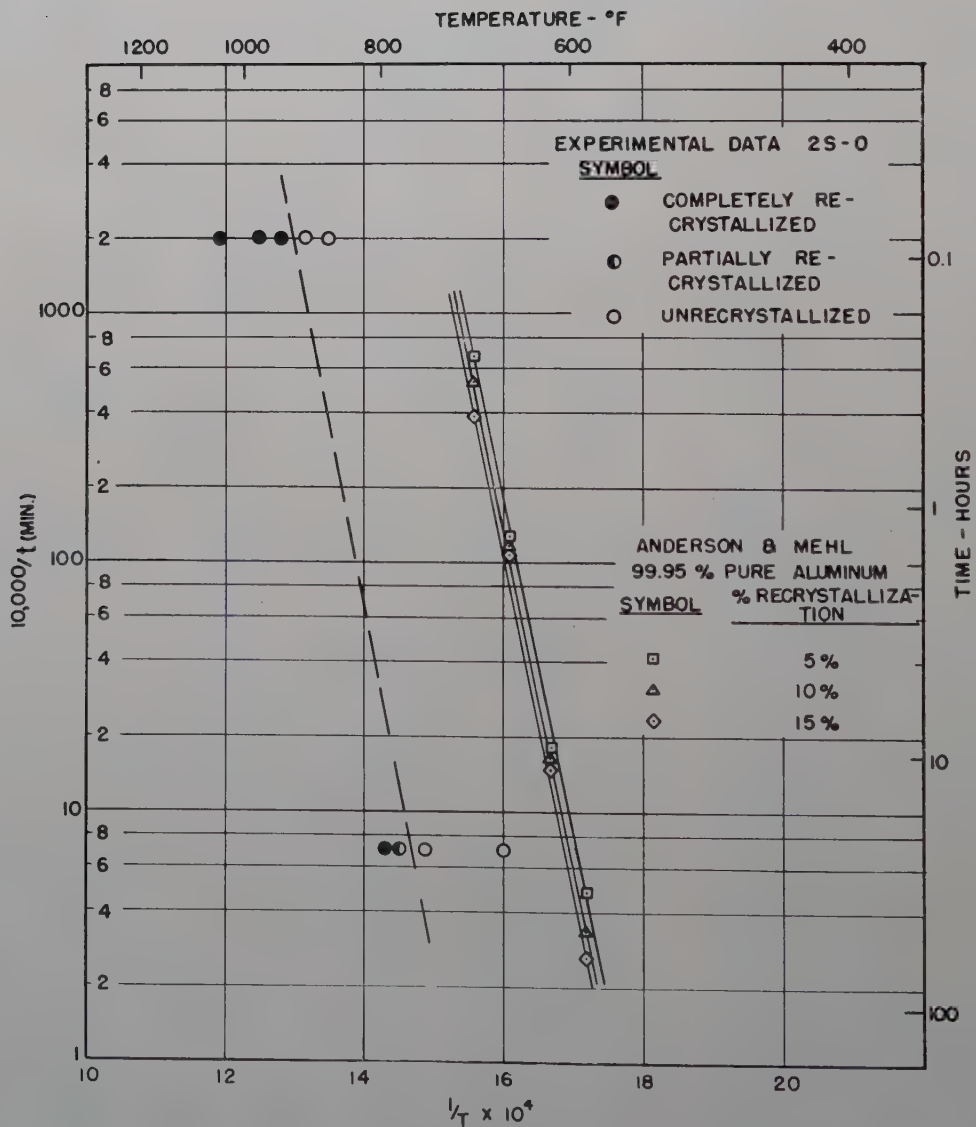


FIG 11—Recrystallization of 2S-O alloy.

of the recovered specimen into coincidence with that for a virgin specimen. Then  $\epsilon_r - \epsilon_u$  shown in Fig 5, would serve as a measure of the strain-recovery. This method should work if only one kind of an imperfection is present. But, as shown in Fig 5, coincidence of the stress-strain curves for the virgin and annealed materials is obtained at only one point, a fact that not only disqualifies the proposed procedure, but also implies that several types of imperfections responsible for work-hardening are induced by cold-work.

In order to obtain a method of measuring the amount of orthorecovery, consider evaluation  $\sigma_u - \sigma_r$  (illustrated in Fig 5) as a function of the total strain. Several typical curves for various recovery treatments are shown in Fig 6. It is immediately apparent that the various curves are substantially parallel. After annealing for 2588 hr at 212°F the lowest curve representing the effect of almost complete metarecovery is obtained. The remaining curves represent the effect of both meta- and orthorecovery for various recovered states. The parallel trends of these curves suggest that metarecovery is almost complete after annealing at 212°F for 2588 hr and that orthorecovery results in a uniform decrease in the flow stress below that for the virgin unrecovered specimen independent of the subsequent strain.

In order to test this concept consider now the data of Fig 7 obtained by prestraining about 0.048 and annealing at 400°F for recovery. If after annealing for 1.5 hr at 400°F all of the metarecovery has taken place as well as some orthorecovery, the difference between the stress-strain curve after 1.5 hr of annealing and that following 1578 hr of annealing is attributable to orthorecovery. Furthermore, if orthorecovery causes a uniform decrease in the flow stress for all strains a vertical shift of the stress-strain curve after annealing 1.5 hr should yield a curve that coincides exactly with that obtained after annealing 1578 hr. Upon shifting the stress-strain curve after 1.5 hr of annealing to lower stress values, the broken curve of Fig 7 is obtained. The excellent agreement between the experimental data and the broken curve support the concept that orthorecovery consists of a uniform lowering of the flow stress for all strains, whereas metarecovery consists in decreasing only the initial flow stress. Consequently,  $\sigma_u - \sigma_r$  at high total strains might be taken as a measure of

orthorecovery.

This concept was also checked and found to give equally good agreement for values of prestrain equal to 0.092 and 0.125. The results of this correla-

tion after a prestrain of 0.125 are shown in Fig 8.

No justification has been presented in the previous discussion in favor of the use of the term orthorecovery. It is

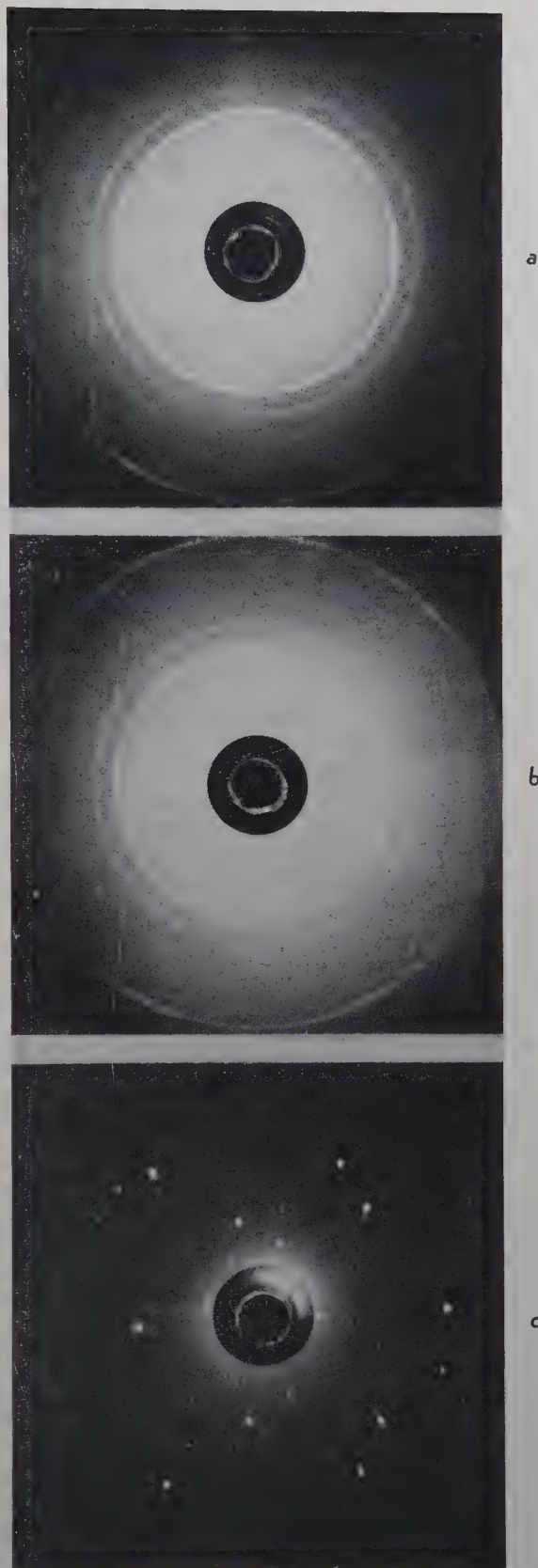


FIG 12—Back reflection X ray patterns. W radiation.



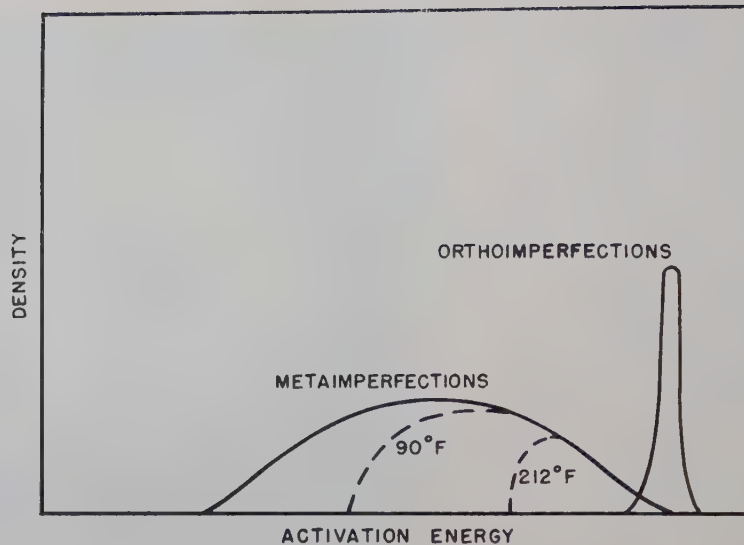


FIG 13—Schematic representation of distribution of imperfections induced by cold work as a function of activation energy for recovery.

quite possible, of course, that the specific phenomena peculiar to what has been called orthorecovery is actually due to recrystallization. Therefore, this possibility was explored.

Mehl et al.<sup>11-14</sup> have demonstrated that the fraction recrystallized-time curve is sigmoidal as illustrated in Fig 9. Although the mechanical properties need not vary linearly with the fraction recrystallized, nevertheless, some sigmoidal relationship would again be expected between the change in flow stress and time of annealing at temperatures of annealing if recrystallization takes place. In order to check this concept, the data for orthorecovery were plotted as a function of time as shown in Fig 9. The value  $\Delta\sigma$  was obtained by taking  $\sigma_w - \sigma_r$  (see Fig 5) at a large total strain ( $\epsilon = 0.24$ ) where metarecovery no longer affects the flow stress. The  $\Delta\sigma$ -time curve of Fig 10 illustrates that the process termed orthorecovery has its greatest rate at  $t = 0$  in sharp contrast to phenomena that depend upon nucleation and growth. This evidence, then, suggests that what has been called orthorecovery is not a true recrystallization process. Furthermore, no detectable difference in microstructure was discernable upon careful comparison of the structures of a specimen strained 0.13 and another strained the same amount and then annealed at 400°F for 1577 hr.

The most conclusive evidence that recrystallization was absent in the annealing treatments leading to ortho-

recovery, however, is shown in Fig 11. It is well known that when the logarithm of the reciprocal of the time to achieve a certain percentage of recrystallization is plotted as a function of the reciprocal of the absolute temperature, a linear relationship, dictated by the laws of kinetics of reaction rates, is obtained. The data recorded by the solid lines of Fig 11 were taken from the excellent investigation of Anderson and Mehl<sup>14</sup> on the recrystallization of high purity aluminum (99.95 pct Al). Thus the linear relationship that is obtained when recrystallization data are plotted in this manner is well established. Now 2S-O alloy is less pure than the material investigated by Anderson and Mehl and, therefore, longer times or higher temperatures of annealing would be required to cause equivalent recrystallization of 2S-O alloy.

A complete investigation of the recrystallization of 2S-O was not attempted, but only an estimate of its recrystallization temperatures and times was made in this study. Specimens of 2S-O alloy were strained 0.09 at atmospheric temperature and annealed for 15 min. and 24 hr respectively at a series of temperatures. Back reflection Laue patterns, using  $W$  radiation, were then taken to establish whether or not recrystallization was obtained. Three typical radiographs are shown in Fig 12a, 12b and 12c after annealing for 24 hr at a series of temperatures. Fig 12a exhibits the continuous rings (due to the  $L$  radiation)

of the fine grained cold-worked 2S-O. Fig 12c illustrates a coarse grained structure after complete recrystallization, whereas Fig 12b shows some rings attributable to the initial fine grain size as well as some spots resulting from the larger recrystallized grains. These data and analogous results for the recrystallization in 15 min. are recorded in Fig 11. Of course, the location of the line representing a given small amount of recrystallization cannot be ascertained accurately from these data. But the approximate curve shown by the broken line is not seriously in error. It is interesting to note that this line is practically parallel to that obtained in the more thorough microscopic investigation on the recrystallization of higher purity aluminum by Anderson and Mehl.

Assuming that the linear relationship of this plot is admissible, the calculated time for incidence of recrystallization of 2S-O alloy prestrained 0.09 and annealed at 400°F is of the order of magnitude of  $10^{10}$  hr. Since the phenomenon described as orthorecovery is already clearly discernable in less than 18 hr after annealing the cold-worked 2S-O alloy, orthorecovery is distinctly separate from a recrystallization process. Orthorecovery appears to be a true recovery process.

Burgers<sup>15</sup> has already discussed the mechanics of the recovery process in terms of dislocations but it will nevertheless be interesting to deduce what kinds of models of work-hardening are consistent with the preceding observa-

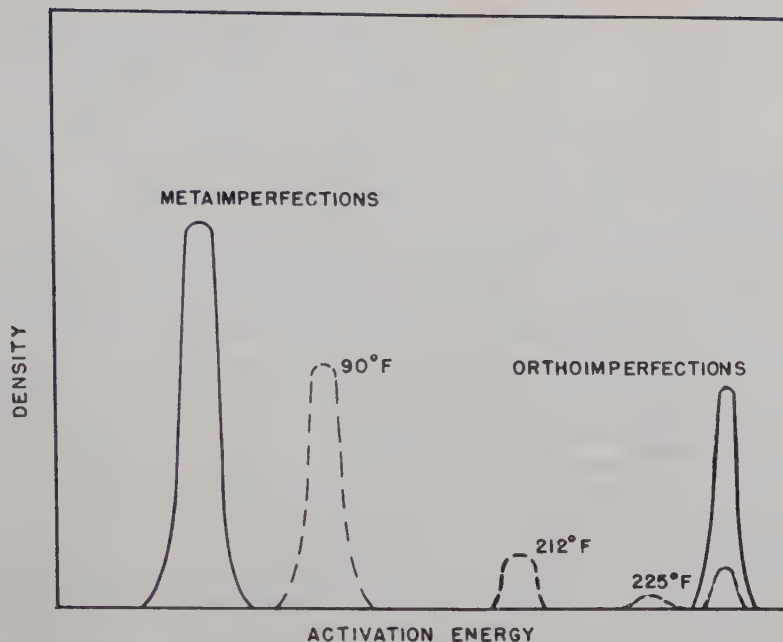


FIG 14—Schematic representation of distribution of imperfections induced by cold work as a function of activation energy for recovery.

tions on the recovery of the flow stress of cold-worked aluminum. It will be convenient to designate those imperfections that are removed by meta-recovery as metaimperflections and those that are removed by pure orthorecovery as orthoimperflections.

The activation energies for the removal of orthoimperflections by recovery must be higher but may overlap the activation energies for annihilation of metaimperflections. The rather uniformly continuing process of orthorecovery suggests that orthoimperflections do not have strong interaction energies and consequently the activation energy for orthorecovery appears to be independent of the density of orthoimperflections.

The process of metarecovery is more complex than that for orthorecovery. First two alternate models appear to be possible. One is shown in Fig 13 based on the assumption that the activation energies for recovery of the metaimperflections consist of a broad band of values and that there is no interaction between the metaimperflections so that their activation energies are independent of their densities. The broken curves marked 90 and 212°F give a schematic representation of the distribution of metaimperflections following several hours recovery at these temperatures. After annealing at 300 and 400°F for a few hours all of the

metaimperflections will have been removed and some of the orthoimperflections will have been eliminated. Since the orthoimperflections exist over a narrow band of activation energies, the process of orthorecovery will continue (but at an ever decreasing rate because of the decrease in the density of orthoimperflections) until complete recovery is achieved.

An alternate model is shown in Fig 14. Here it is assumed that the metaimperflections have an activation energy for recovery that is a function of their density. As they are removed by metarecovery their activation energy increases as shown by the broken curves which represent the metastable states achieved after annealing a few hours at 90 and 212°F respectively. At temperatures of 300 and 400°F recovery of all of the metaimperflections occurs in a very short period of time and thereafter pure orthorecovery continues, the orthoimperflections being eliminated without major changes in the activation energies of those that remain.

It is helpful now to define more precisely the kinetics of the recovery process. To a first approximation the process might be considered analogous to a unimolecular reaction wherein the rate of recovery is proportional to the number of imperfections per unit volume, namely

$$-\frac{dN}{dt} = kN$$

where  $N$  is the density of dislocation,  $t$  is the time and  $k$  is the rate constant. For metarecovery the rate constant  $k$  is probably given by

$$k = k_0 e^{-\{E-f(N)\}/RT}$$

where  $f(N)$  is some function of the density of imperfections,  $E$  is the activation energy,  $R$  the gas constant and  $T$  the absolute temperature.

Thus, as recovery takes place  $E - f(N)$  increases and the process substantially ceases before complete recovery is achieved. Unfortunately the present evidence on metarecovery rates is too incomplete to evaluate  $f(N)$ .

For orthorecovery, however, no interaction energy is assumed and  $k$  is simply given by

$$k = k_0 e^{-E/RT}$$

Consequently

$$\ln \left( \frac{Ni}{N} \right) = kt = tk_0 e^{-E/RT}$$

where  $Ni$  is the number of orthoimperflections at  $t = 0$ . Then

$$\ln \ln \left( \frac{Ni}{N} \right) = \ln k_0 - \frac{E}{RT} + \ln t$$

Assuming that when  $\sigma_u - \sigma_r$  reaches some value upon orthorecovery,  $Ni/N$  is constant, the activation energy  $E$  is obtainable by plotting  $\ln t$  vs.  $1/T$  for constant values of  $\sigma_u - \sigma_r$ . The data for orthorecovery reported here for 2S-O alloy reveal that  $E$  is a constant



independent of the time of recovery. Furthermore,  $E$  is about 33,000 cal per mol. It is scarcely accidental that this value agrees well with the activation energies in Table 1 quoted by Zener.<sup>16</sup>

According to either of the postulated mechanisms, orthorecovery is not compatible with the recovery of Taylor's patterns of dislocations, whereas meta-recovery of the kind illustrated in Fig 13 is not inconsistent with Taylor's concept of the work-hardened state.

The assumption that work-hardening introduced two distinctly different types of imperfections, namely the meta- and the orthoimperfections, not only facilitates the analysis of the recovery data reported here, but it also clarifies several issues of long standing regarding the recovery process. First, the models for recovery based on the presence of meta- and orthoimperfections demand that the work-hardened state, after partial recovery, be dissimilar from any state achieved by work-hardening alone. This conclusion is necessary for at the lower annealing temperatures only the metaimperfections are eliminated whereas both imperfections are introduced simultaneously upon strain-hardening. Consequently, recovery is not exactly the reverse of strain-hardening. Secondly, it now becomes clearer why properties exhibit unique recovery rates upon annealing. Some properties, such as the yield strength, (and perhaps the electrical resistivity) are sensitive to recovery of metaimperfection whereas other properties, such as tensile strength, are rather insensitive to the metaimperfections and respond most effectively to the recovery of the orthoimperfections. Consequently, some properties may exhibit almost complete recovery before any appreciable recovery is detectable in other properties.

## Conclusions

1. After recovery at low tempera-

**Table 1 . . . Activation Energies for Aluminum**

Process	
Volume Diffusion <sup>16</sup> .....	37,500
Grain Boundary Slip <sup>16</sup> .....	34,500
Creep <sup>16</sup> .....	37,000
Orthorecovery.....	33,000

tures (90 and 212°F) cold-worked aluminum exhibits a low initial flow stress upon restraining. But after an additional strain of about 0.04 the flow stress is restored to essentially that value which would have been obtained if the specimen had not been recovered. This is called metarecovery.

2. At higher temperatures (300 and 400°F) not only is metarecovery observed, but a permanent decrease in the flow stress is obtained such that the stress-strain curve for the recovered metal is below that for the virgin metal at the same total strain for all strains. This is called orthorecovery.

3. The kinetics of meta- and orthorecovery appear to be distinctly different. This suggests that during work-hardening at least two kinds of imperfections are introduced, one which is rapidly recoverable, and a second which is more slowly recoverable. This assumption rationalizes the observed differences in recovery rates of different properties.

4. These observations strongly suggest that the work-hardened state is characterized by at least two essentially distinct types of imperfections.

## Acknowledgment

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towski for the preparation of the figures.

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# Carbides in Isothermally Transformed Chromium Steels

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Electrolytic extraction of carbides from quenched and tempered steel and their examination under the electron microscope were found to be helpful in understanding the mechanism of secondary hardening in alloy steels<sup>1</sup> and the same technique has been applied to isothermally transformed steels. A preliminary survey of the utility of the method has indicated that it has promising possibilities for investigating the characteristics of pearlite and bainite. The examination of a few carbon and chromium steels has suggested that pearlite is formed with carbides of two varieties representing the lamellar and nonlamellar forms and that bainite appears to form with a structure analogous to, but significantly different from, martensite.

## Procedure

The method used for examining the carbides was similar to that described previously.<sup>1</sup> The chromium steels used in the investigation were made from an Armco iron base in an induction furnace and were not treated with grain-refining deoxidizers. The composition of the steels is given in Table 1.

Three-inch square ingots were forged and rolled to  $\frac{1}{8}$ -in. thick sheet for use in the isothermal studies. After rolling, the sheets were sandblasted to remove the rolling scale.

Specimens approximately  $\frac{3}{4}$ -in. wide and  $1\frac{1}{2}$ -in. long were prepared from the  $\frac{1}{8}$ -in. sheet. They were heated in a salt bath at 2100°F (1150°C) for  $\frac{1}{2}$  hour, transferred to other salt baths at 1300°F (704°C), 1000°F (538°C), 800°F (426°C), or 600°F (315°C), held

Table 1 . . . Composition of Steels

Steel	Pct C	Pct Mn	Pct Si	Pct Cr
A	0.20	0.55	0.20	5.06
B	0.43	0.52	0.23	5.41
C	1.17	0.43	0.33	5.95

for various periods up to 100 hr and finally quenched in water. After quenching, the specimens were cut in half, one half being submitted to microscopic examination and the other half being used for the electrolytic extraction. The isothermally transformed specimens were submitted to examination under the light microscope to determine the degree of transformation and type of structure and were photographed at 2000 $\times$ .

Carbide residues were obtained by electrolyzing in 10 pct hydrochloric acid and collecting the residue in glycerine to minimize attack by the acid. As pointed out in the earlier paper, the carbides are attacked to some degree during electrolysis. This attack is relatively minor on the coarser carbides but may be quite severe on the fine bainitic carbides. The residues were washed free of the

glycerine with water and alcohol. The alcohol was removed by repeated washings of amyl acetate, and the amyl acetate suspensions were transferred to stoppered vials. Debye-Sherrer X ray diffraction patterns of the residues were prepared using a chromium target. A selection based on the microstructure and X ray data was then made of samples considered most illustrative of the progress of transformation. Electrolytically extracted residues from these samples were submitted to examination by W. D. Forgeng and A. C. Jenkins on the electron microscope at the Research Laboratories of The Linde Air Products Co. and electron micrographs were made at  $\times 5000$  and enlarged to 25,000 $\times$ . The micrographs obtained with both the light and the electron microscopes are mounted side by side in the accompanying figures.

Although no effort was made to establish the T-T-T diagram for the chromium steels, the times for holding in the salt bath were adjusted with the intention of making the samples represent different stages in the transformation at temperatures of 600, 800, 1000, and 1300°F. The degrees of transformation and results of X ray examination are shown in Tables 2-4. The carbide designations refer to orthorhombic or trigonal carbide types and are not meant to imply that the compositions are exactly as indicated.

## X ray Diffraction Data

The occurrence of  $\text{Fe}_3\text{C}$  and  $\text{Cr}_7\text{C}_3$  in these steels was found to be consistent with published data on comparable chromium steels. Steels A and B with relatively low ratios of carbon to chromium contained  $\text{Cr}_7\text{C}_3$  in the pearlitic structures and  $\text{Fe}_3\text{C}$  in the

New York Meeting, February 1950.

TP 2709 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before May 1, 1950. Discussion is tentatively scheduled for publication in November 1950.

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<sup>1</sup> References are at the end of the paper.



**Table 2 . . . Constituents in X Ray Patterns of Extracted Residues of Steel A—0.20 Pct C—5.06 Pct Cr After Indicated Degree of Transformation**

1300°F						
Time at Temp.	36 Sec	6 Min	20 Min	1 Hr	10 Hr	100 Hr
Pct transformation.....	0	1-2*	15	99*	99	100*
X ray pattern.....	(a)	Blank	Cr <sub>7</sub> C <sub>3</sub> (3) Cr <sub>2</sub> N (2) p (b) (1)	Cr <sub>7</sub> C <sub>3</sub> (3) Cr <sub>2</sub> N (1) p (b) (4)	Cr <sub>7</sub> C <sub>3</sub> (4) Cr <sub>2</sub> N (4)	Cr <sub>7</sub> C <sub>3</sub> (4) Cr <sub>2</sub> N (4)
1000°F						
Pct transformation.....	0	0		0	0	1*
X ray pattern.....	(a)	(a)		(a)	(a)	Blank
800°F						
Pct transformation.....	1-2	85*		90	95	98*
X ray pattern.....	Blank	Blank		Fe <sub>3</sub> C (2)	Fe <sub>3</sub> C (2)	Fe <sub>3</sub> C (2)

- \* Residues submitted to electron microscope studies.  
(a) No extractions made.  
(b) Unidentified constituent—For *d*-values see Table 5.  
(1) Very faint pattern.  
(2) Poor pattern.  
(3) Fair pattern.  
(4) Good pattern.

**Table 3 . . . Constituents in X Ray Patterns of Extracted Residues of Steel B—0.43 Pct C—5.41 Pct Cr After Indicated Degree of Transformation**

1300°F						
Time at Temp.	6 Min	½ Min	12 Hr	1 Hr	10 Hr	100 Hr
Pct transformation.....	5	20*	95	98*	99	100*
X ray pattern.....	Blank	Cr <sub>7</sub> C <sub>3</sub> (2) p (b) (1)	Cr <sub>7</sub> C <sub>3</sub> (3) p (b) (2)	Cr <sub>7</sub> C <sub>3</sub> (3) p (b) (4) Cr <sub>2</sub> N (1)	Cr <sub>7</sub> C <sub>3</sub> (4) Cr <sub>2</sub> N (4)	Cr <sub>7</sub> C <sub>3</sub> (4) Cr <sub>2</sub> N (4)
1000°F						
Pct transformation.....	0			0	1	35*
X ray pattern.....	(a)			(a)	Blank	Cr <sub>7</sub> C <sub>3</sub> (4) p (b) (4)
600°F						
Pct transformation.....	1		20*	75	80*	95
X ray pattern.....	Blank		Fe <sub>3</sub> C (1)	Fe <sub>3</sub> C (3)	Fe <sub>3</sub> C (4)	Fe <sub>3</sub> C (4)

- \* Residues submitted to electron microscope studies.  
(a) No extractions made.  
(b) Unidentified constituent—For *d*-values see Table 5.  
(1) Very faint pattern.  
(2) Poor pattern.  
(3) Fair pattern.  
(4) Good pattern.

**Table 4 . . . Constituents in X Ray Patterns of Extracted Residues of Steel C—1.17 Pct C—5.95 Pct Cr After Indicated Degree of Transformation†**

1300°F						
Time at Temp	3.25 Min	6 Min	1 Hr	3 Hr	10 Hr	100 Hr
Pct transformation.....	65*	95	99*		99	100*
X ray pattern.....	Cr <sub>7</sub> C <sub>3</sub> (2) Fe <sub>3</sub> C (3)	Cr <sub>7</sub> C <sub>3</sub> (3) Fe <sub>3</sub> C (3)	Cr <sub>7</sub> C <sub>3</sub> (4) Fe <sub>3</sub> C (3) p (b) (4)		Cr <sub>7</sub> C <sub>3</sub> (4) Fe <sub>3</sub> C (3) p (b) (1)	Cr <sub>7</sub> C <sub>3</sub> (4)
1000°F						
Pct transformation.....		Trace	5*	50*	95	98*
X ray pattern.....		Cr <sub>7</sub> C <sub>3</sub> (2)	Cr <sub>7</sub> C <sub>3</sub> (2) Fe <sub>3</sub> C (2)	Cr <sub>7</sub> C <sub>3</sub> (3) Fe <sub>3</sub> C (3) p (b) (4)	Cr <sub>7</sub> C <sub>3</sub> (3) p (b) (4)	Cr <sub>7</sub> C <sub>3</sub> (4) p (b) (4)
600°F						
Pct transformation.....		0	0		1	5*
X ray pattern.....		(a)	(a)		Cr <sub>7</sub> C <sub>3</sub> (3)	Cr <sub>7</sub> C <sub>3</sub> (2) Fe <sub>3</sub> C (2)

- \* Residues submitted to electron microscope studies.  
† Cr<sub>7</sub>C<sub>3</sub> noted in X ray pattern of direct quenched sample.  
(a) No extractions made.  
(b) Unidentified constituent—For *d*-values see Table 5.  
(1) Very faint pattern.  
(2) Poor pattern.  
(3) Fair pattern.  
(4) Good pattern.

bainitic range. A pattern of the Cr<sub>2</sub>N type was also found in steels A and B transformed at 1300°F. In general, the patterns of Fe C, Cr<sub>7</sub>C<sub>3</sub>, and Cr<sub>2</sub>N increased in definition and intensity with the length of time the specimens were held at temperature.

Undissolved carbide was present after austenitizing steel C and X ray diffraction revealed a corresponding Cr<sub>7</sub>C<sub>3</sub> pattern in all specimens. The carbide formed as the initial product of transformation at all temperatures appeared to be Fe C, but this pattern tended to disappear from pearlitic structures, leaving only Cr<sub>7</sub>C<sub>3</sub> after long holding at temperature. As shown in Table 5, the dimensions of the

**Table 5 . . . *d*-Values of Cr<sub>7</sub>C<sub>3</sub> Type Carbides and Unidentified Constituent in 5 Pct Chromium Steel Residues**

Cr <sub>7</sub> C <sub>3</sub> Type from Steels A and B		Cr <sub>7</sub> C <sub>3</sub> Type from Steel C		Unidentified Constituent	
<i>d</i> -Å	Relative Intensity	<i>d</i> -Å	Relative Intensity	<i>d</i> -Å	Relative Intensity
2.28	5	2.27	5	2.43*	2
2.11	5	2.10	6	2.08	3
2.03	10	2.02	10	2.06	2.5
2.01	2	1.99	2	1.93	1.5
1.89	1	1.90	1	1.59	2
1.84	2	1.82	3	1.23	1.5
1.80	4	1.79	5		
1.74	3	1.73	3		
1.60	0	1.59	1		
1.43	2	1.42	3		
1.34	2	1.34	3		
1.32	1	1.315	2		
1.25	1	1.245	1		
1.199	4	1.195	5		
1.175	4	1.172	5		
1.163	3	1.159	3		
1.159	3	1.154	3		

\* Not known if this value is a result of this constituent.

Cr<sub>7</sub>C<sub>3</sub> pattern found in the 1.17 pct carbon steel C were different from those in the Cr<sub>7</sub>C<sub>3</sub> pattern found in the lower carbon steels A and B. The Cr<sub>7</sub>C<sub>3</sub> pattern of a steel containing 0.70 pct carbon and 5 pct chromium was also of the same dimensions as that found in the lower carbon steels, so that the change would seem to be discontinuous and probably represents a decided difference in composition.

In addition to the normally expected phases, the residues of specimens held for intermediate periods at temperatures producing pearlitic structures also contained a phase that gave a pattern with "*d*" values as given in Table 5. The pattern of this phase tended to appear only after the transformation was well advanced and to disappear on holding after completion of transformation. The nature of the phase has not been identified.

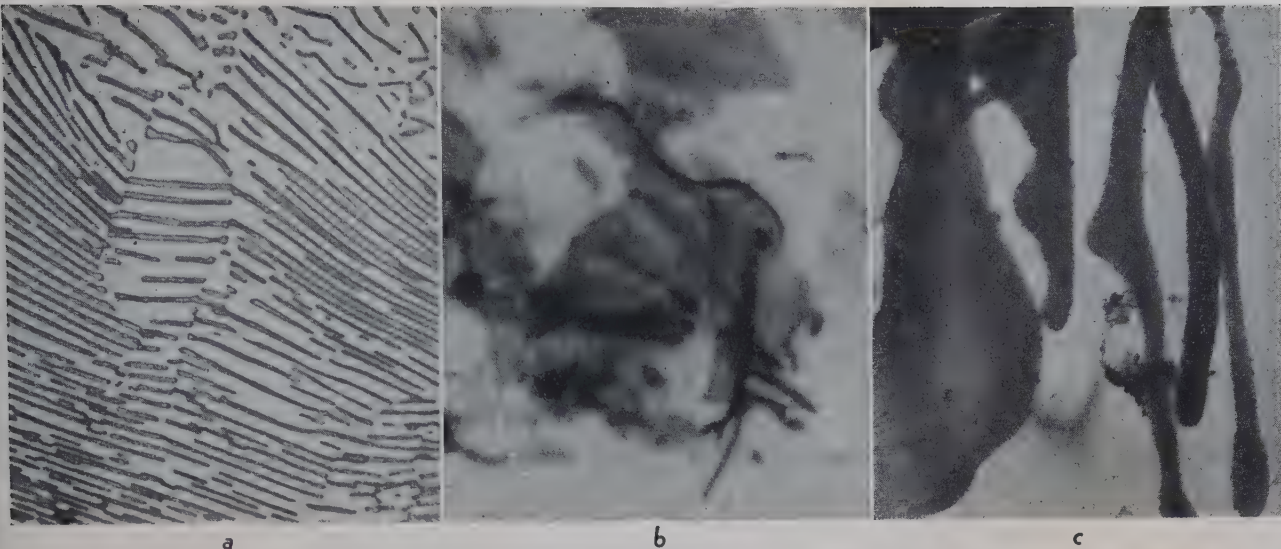


FIG 1—Eutectoid carbon steel, cooled very slowly from 1850°F.

A—Micrograph of steel.  $\times 2000$ . B—Micrograph of extracted residue.  $\times 2000$ . C—Electron micrograph of extracted residue.  $\times 20,000$ .

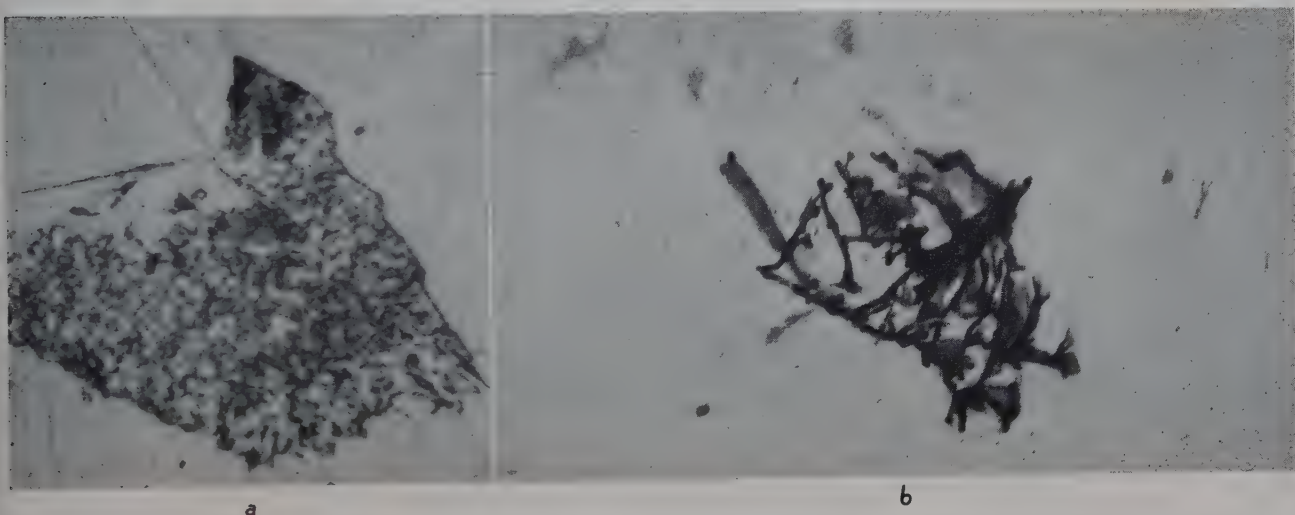


FIG 2—Steel A—0.20 pct C, 5.06 pct Cr. Transformed 1–2 pct after 6 min at 1300°F.

A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .

## Microscopic Examination of Carbides in Pearlite

A limited number of carbon steels, transformed isothermally and by continuous cooling, as well as the chromium steels, have been studied by electrolytic extraction and examination of the carbide residue on both the light and electron microscopes. Only the coarser structures permit a direct comparison at comparable magnification between the structure found in the metal and the appearance of extracted carbides as revealed by transmitted light and the electron beam. However, the structures are sufficiently similar to make the higher magnification electron micrographs of finer structures quite convincing.

The pearlitic structures found in the 5 pct chromium steels were quite fine,

only partly lamellar, and generally of a confused and granular appearance rather than the well defined lamellar structure of slowly cooled carbon steel. The carbides extracted from well developed lamellar pearlite in a slowly cooled eutectoid carbon steel are illustrated by light and electron micrographs in Fig 1. The typical carbide is a thin, very irregularly shaped, and often curled plate. It may contain holes and usually has deeply indented bays. There are thickened knobs and fingers at the edges and sometimes within the body of the plate. The thickened edges appear to correspond with the clubby ends of pearlite lamellae seen in polished cross-sections of steel. This type of carbide was illustrated by Koch<sup>2</sup> using a similar procedure.

The predominant carbides extracted

from the fine pearlite of carbon steels transformed at relatively low temperatures are thicker skeletal or rod-like shapes of very irregular appearance. This type of carbide is illustrated in many of the micrographs of chromium steel residues where it will be seen that it is too thick to be penetrated by the electron beam. Because the thin plate type is predominant in specimens with well developed lamellar pearlite and the more massive skeletal type occurs mainly in specimens with little of the lamellar structure, it would appear that confused pearlite structures are actually not lamellar, but are composed mainly of carbides of the massive skeletal type. Both types of carbide are specific to pearlitic structures and may be readily distinguished from the residues of bainitic or tempered martensitic structures.



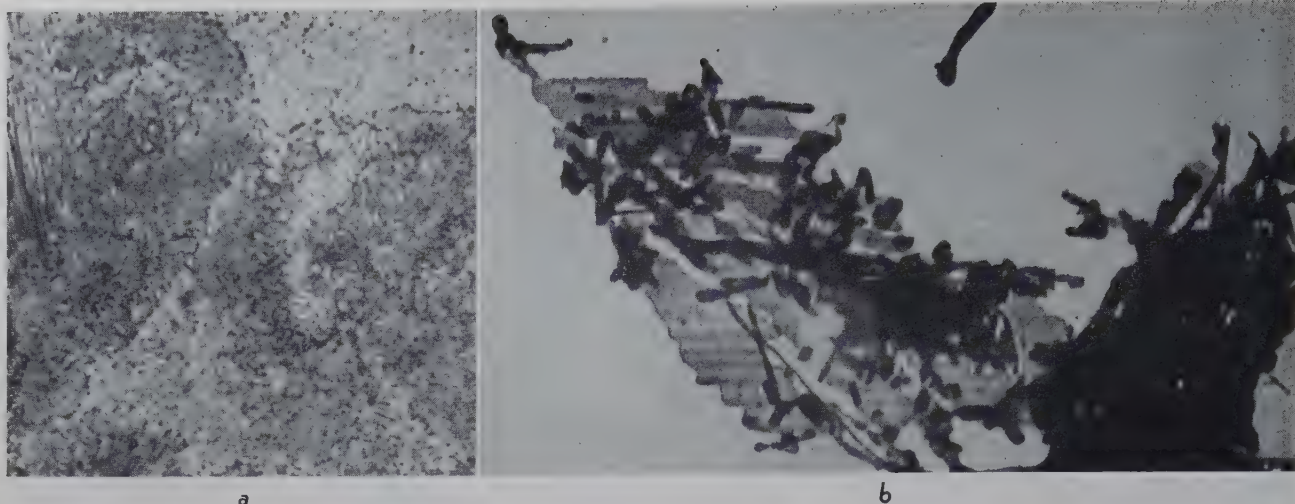


FIG 3—Steel A—0.20 pct C, 5.06 pct Cr. Transformed 99 pct after 1 hr at 1300°F.  
A—Steel structure.  $\times 2000$ . B—Extracted residue, Electron micrograph.  $\times 25,000$ .

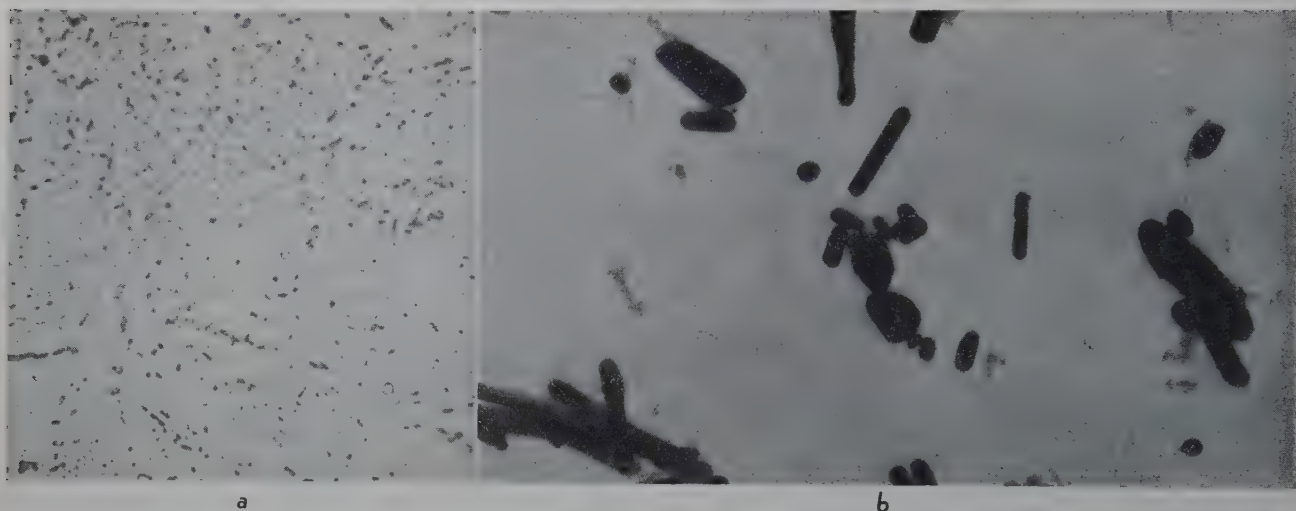


FIG 4—Steel A—0.20 pct C, 5.06 pct Cr. Transformed 100 pct after 100 hr at 1300°F.  
A—Steel structure.  $\times 2000$ . B—Extracted residue, Electron micrograph.  $\times 25,000$ .

The carbides extracted from the pearlitic chromium steels, as well as micrographs of the corresponding steel structures are shown in Fig 2-5, 8-12, 14-19, and 21-22.

The carbides found in steel A transformed at 1300°F, as shown in Fig 2, show an initially fine structure composed mainly of the massive type of carbide in a complex irregular pattern. After 1 hr at 1300°F, as shown in Fig 3, the transformation was virtually complete, and the carbide particles are much coarser than in Fig 2. Both thin and thick carbides are present in a much more well developed form. The thin plate is seen to be corrugated or ribbed to a degree that was not observed in carbon steels, and the massive types are more crystalline in shape than at earlier stages in the transformation. After 100 hr at 1300°F, as shown in Fig 4, the light microscope revealed a spheroidized structure with virtually no lamellae, and the extracted residue

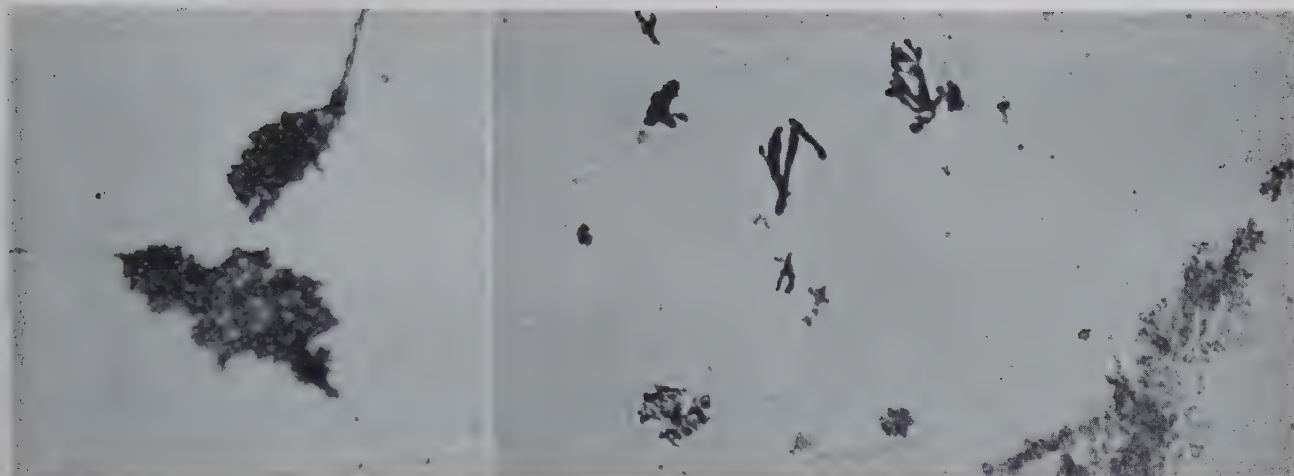
contained only massive, more or less idiomorphic, crystals. The thin plates were absent and had either been absorbed or had coalesced into the idiomorphic carbides. Similar carbide particles were shown by Koch and Wiester<sup>3</sup> after an extended tempering of pearlite.

Both thin and massive carbides in steel B transformed at 1300°F, as shown in Fig 8-10, tend to be very long and relatively narrow, but are qualitatively similar to those found in steel A. The tendency toward thickening at the edges of the thin plates is particularly well illustrated in Fig 8. Some of the thin carbides are still present after 100 hr, although the predominant type is the well developed crystals. The appearance of the thin carbide shown in Fig 10 is somewhat more dense than is typical of specimens held for a shorter time at temperature, and there is a tendency toward local thickening. It is presumed that this represents a

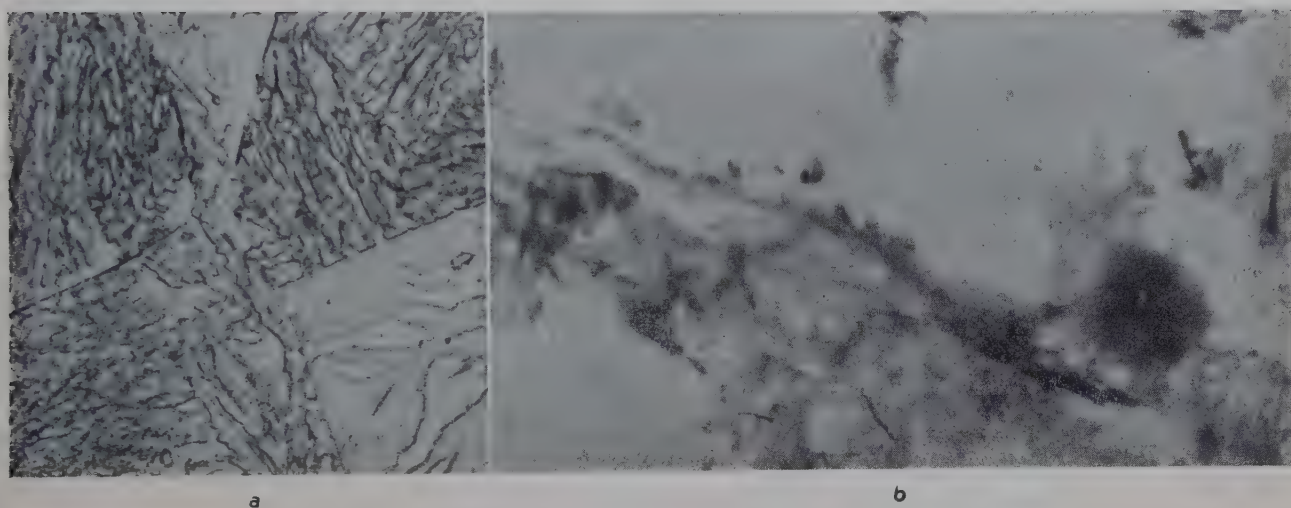
typical mechanism by which the thin particles are converted to a more massive form, although the time involved is so great that a good deal of absorption and reprecipitation could occur. It is probable that the growth of the massive particles also progresses by a diffusion reaction.

The carbides found in steels A and B after 100 hr at 1000°F, Fig 5 and 11, are appreciably smaller than the carbides found after a comparable degree of transformation at 1300°F. However, they are quite similar in character even though the structure as seen in the light microscope is too fine to be resolved satisfactorily.

The carbides produced at 1300°F in steel C, as shown in Fig 14-16, are quite similar to those found in steel A. Although some undissolved carbide was present in these samples, it was not observed in the extracted residues. Ordinarily large carbide particles are too heavy to be supported by the suspending



**FIG 5—Steel A—0.20 pct C, 5.06 pct Cr. Transformed 1 pct after 100 hr at 1000°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 6—Steel A—0.20 pct C, 5.06 pct Cr. Transformed 85 pct after 6 min at 800°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .

ing film used for electron microscope examination, so that they may not be observed in the examination of extracted residues. Transformation of steel C at 1000°F produced “arborescent” pearlite structures as shown in the light micrographs of Fig 17–18. Although the extracted carbides are quite small in the initial stages of transformation, they do not appear to be significantly different from those formed at 1300°F. In the same manner as at higher temperatures, the carbides become progressively larger as the specimens are held longer at temperature.

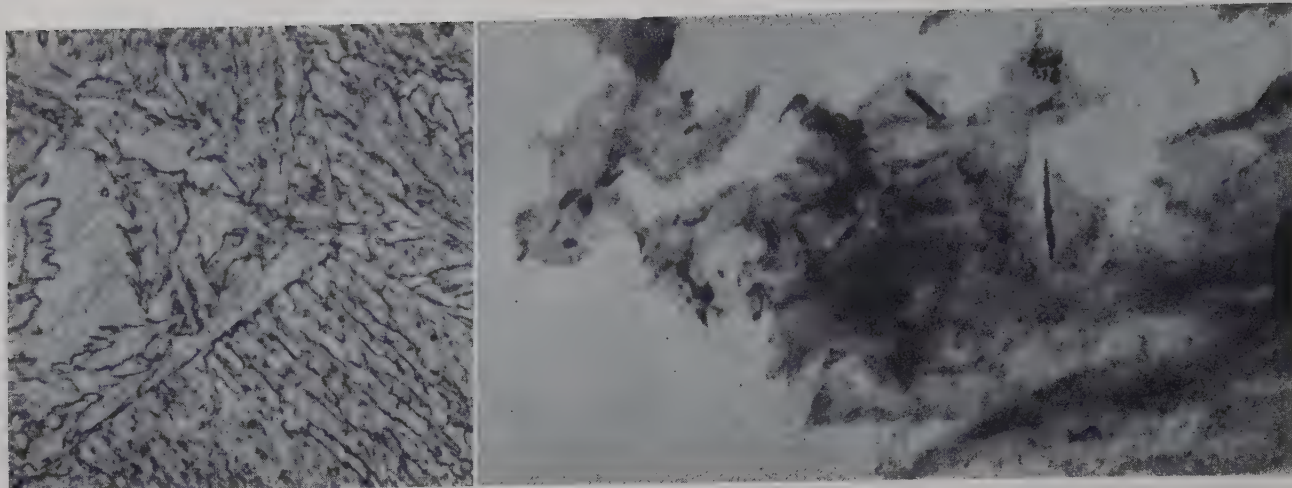
In general, it would appear that the carbide particles in pearlite are of a similar character regardless of the specific composition of the steel or temperature of transformation. There are two well defined types of carbide shape. The difference in character between the thin plates and the more massive carbide particles is shown in Fig 21. The extracted residue was

shadowed with gold before examination on the electron microscope, and the resulting electron micrographs clearly show the relative thickness of the carbide particles. One is a thin plate of very irregular shape tending toward heavier knobs and fingers at the edges. The other is a more massive type that occurs in an irregular skeletal or rod-like form. Both types eventually grow into the idiomorphic form illustrated in Fig 22. The thin type occurs more commonly in well organized lamellar structures and the more massive skeletal type tends to be formed in the more confused sublamellar structures. It would seem reasonable to believe that the plates represent the carbides of strictly lamellar pearlite. Pearlite in which it is difficult to see lamellae is presumably composed principally of the more massive skeletal type of carbides. It would appear that those nonlamellar areas of pearlite that are sometimes considered to be oriented

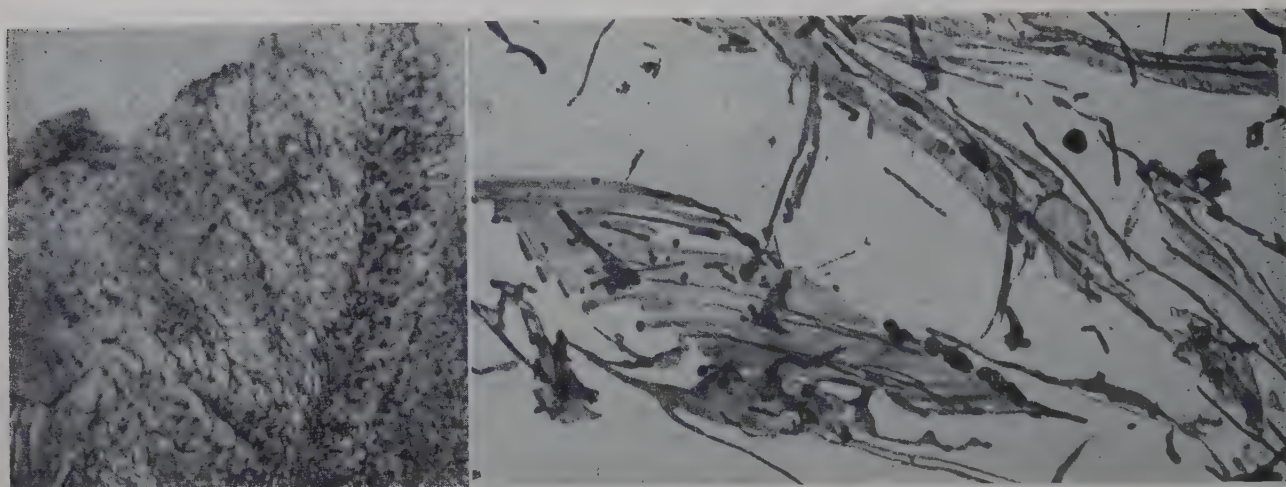
unfavorably to visualize lamellae are actually nonlamellar.

A tempering process with growth of the carbide particles seems to start immediately after transformation and to progress continuously before and after transformation is completed. It was not possible to isolate small carbide particles in the later stages of transformation and identify them as having been formed more recently than the larger carbides. Because of the time involved in the later stages of transformation, it is considered probable that even the freshly formed carbides would be tempered appreciably before examination. It is doubted, therefore, whether it would be reasonable to expect that this method of examination will readily reveal whether the size of carbides transformed late are affected by the growth of previously formed carbides. The progress of tempering of both the thin plates and the skeletal carbides to larger massive and ultimately

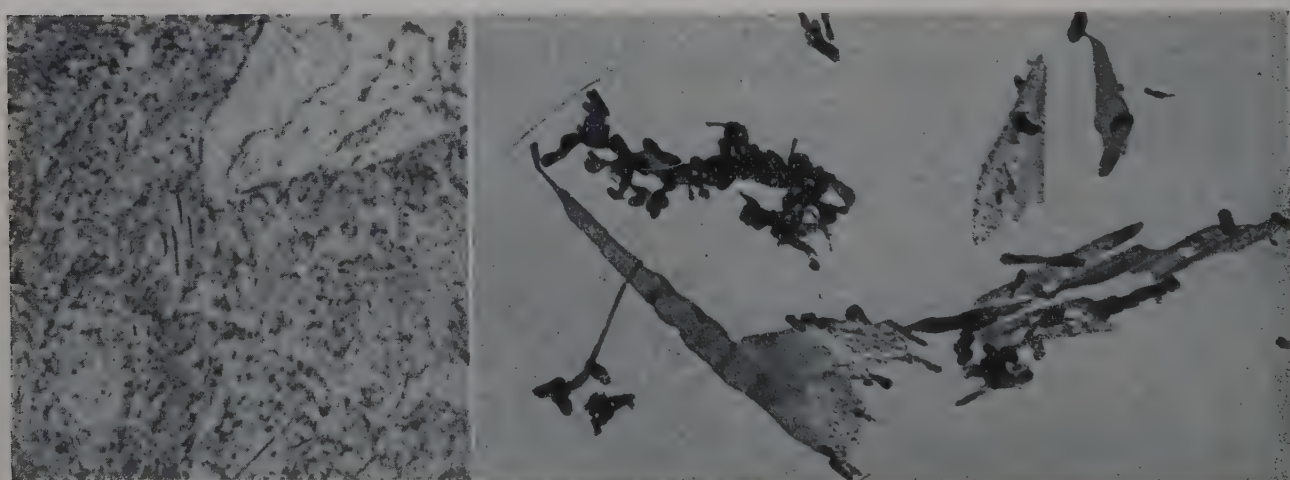




**FIG 7—Steel A—0.20 pct C, 5.06 pct Cr. Transformed 98 pct after 100 hr at 800°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 8—Steel B—0.43 pct C, 5.41 pct Cr. Transformed 20 pct after 12 min at 1300°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 9—Steel B—0.43 pct C, 5.41 pct Cr. Transformed 98 pct after 1 hr at 1300°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .

idiomorphic particles seems to be accomplished in part by coagulation of the more extended shapes as well as by normal diffusion and growth.

### Microscopic Examination

### of Residues from Bainitic Structures

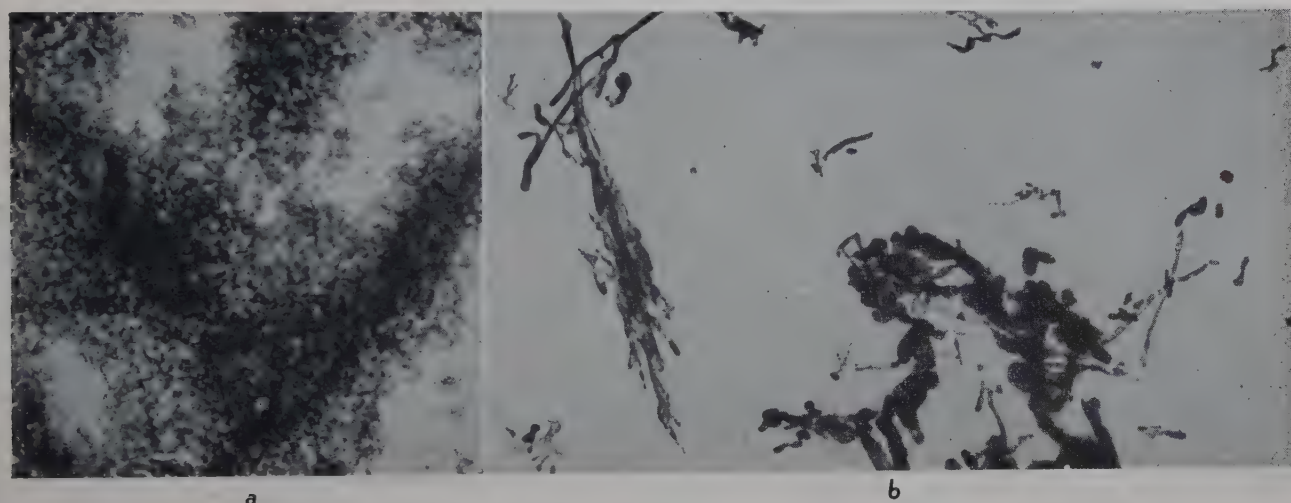
The structures formed in chromium steel specimens treated to produce bainite are illustrated in Fig 6-7, 12-13,

20 and 23. The structures of the extracted residues appear to consist of a nebulous cloud of very fine particles in which there is a concentration of many or larger particles on certain planes as well as a number of well defined par-

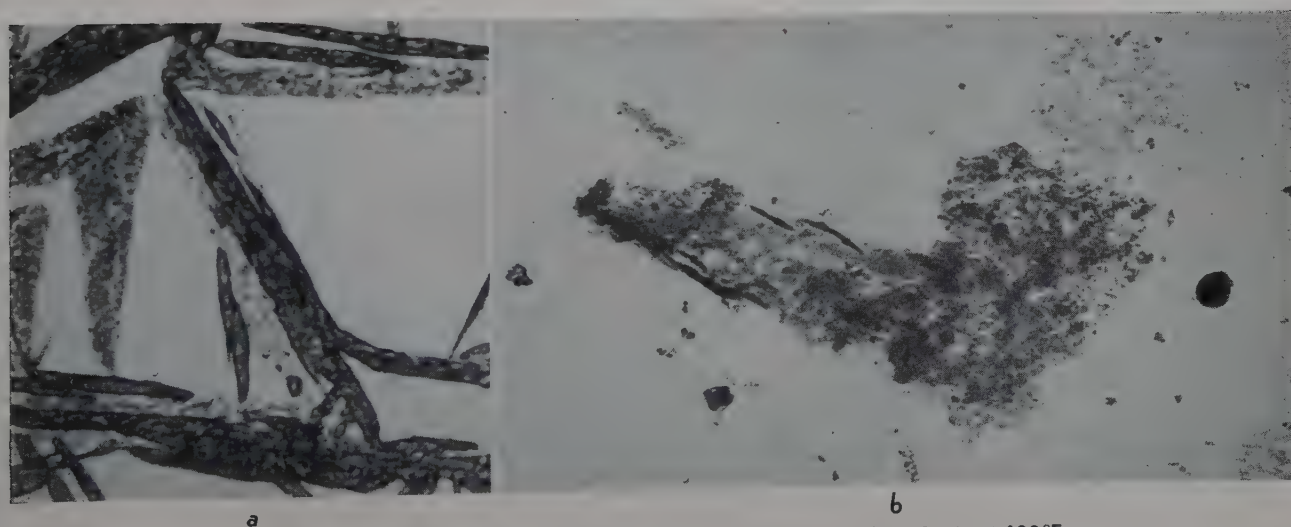




**FIG 10—Steel B—0.43 pct C, 5.41 pct Cr. Transformed 100 pct after 100 hr at 1300°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 11—Steel B—0.43 pct C, 5.41 pct Cr. Transformed 35 pct after 100 hr at 1000°F.**  
A—Steel Structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 12—Steel B—0.43 pct C, 5.41 pct Cr. Transformed 20 pct after 30 min at 600°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .

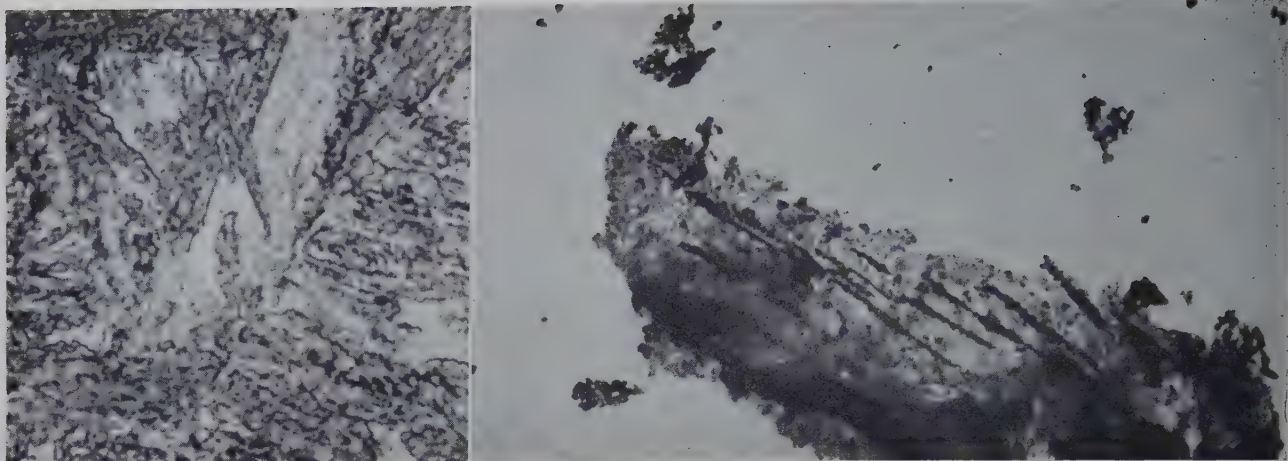
ticles of a larger order of size. The "graininess" or size of the particles seems to increase as time at temperature and degree of transformation increase, but the structure never becomes sufficiently well defined to give

much assurance that the structures are adequately resolved.

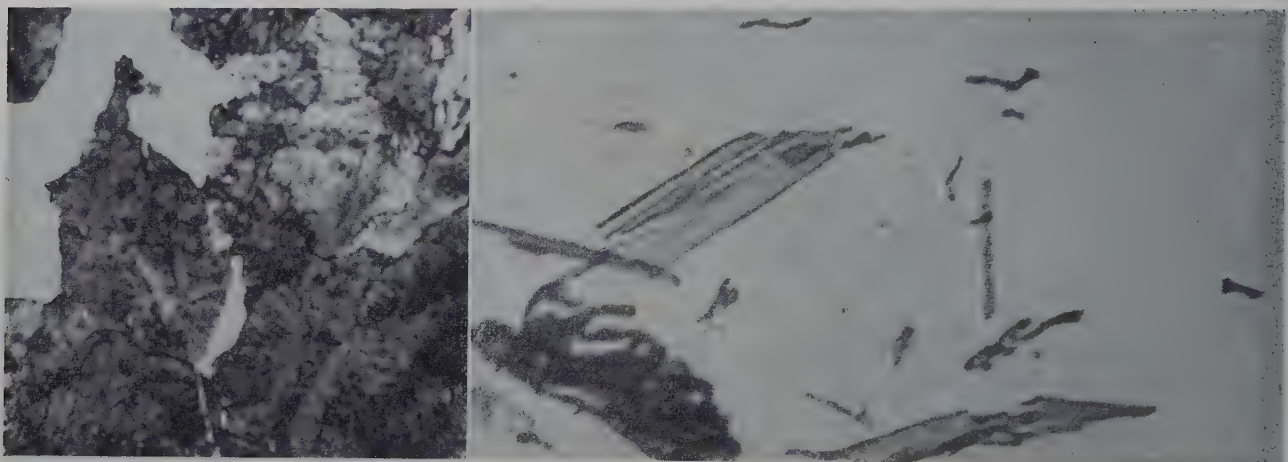
Each group or cloud that appears to be a cluster of a myriad of particles has been shown by gold shadowing in Fig 23 to have a considerable thickness,

so that the electron beam has partially penetrated a great volume of fairly low density. The velvety black zones in Fig 6 and 20 are typical of residues extracted from martensite in which it is probable that the residue itself is only

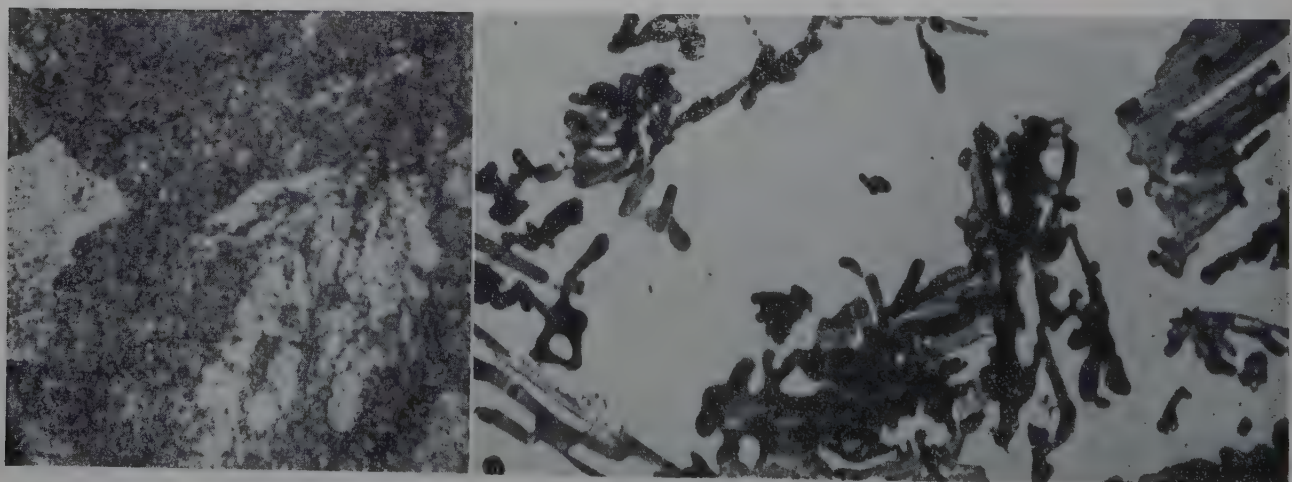




**FIG 13—Steel B—0.43 pct C, 5.41 pct Cr. Transformed 80 pct after 10 hr at 600°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 14—Steel C—1.17 pct C, 5.95 pct Cr. Transformed 65 pct after 3.25 min at 1300°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



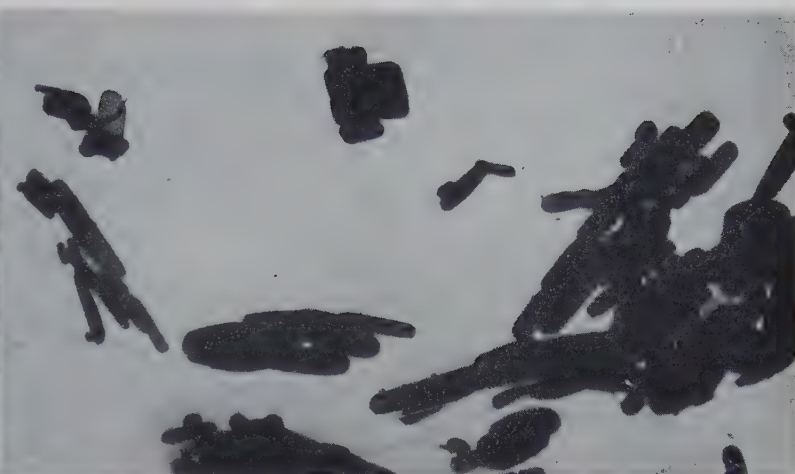
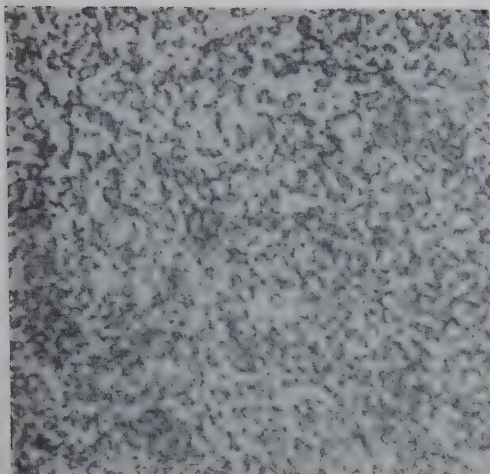
**FIG 15—Steel C—1.17 pct C, 5.95 pct Cr. Transformed 99 pct after 1 hr at 1300°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .

amorphous carbon. No analytical work has been done on these bainite residues, so that it is possible that the residue is not carbide but a metamorphosed representation. The granular structure of the "clouds" is quite different from

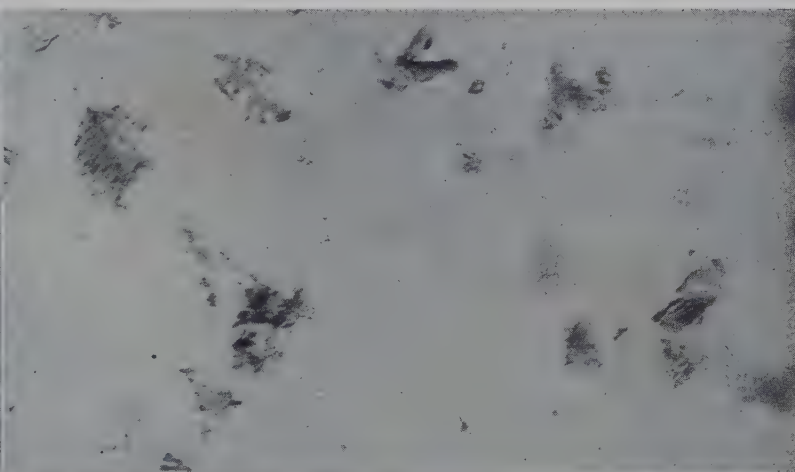
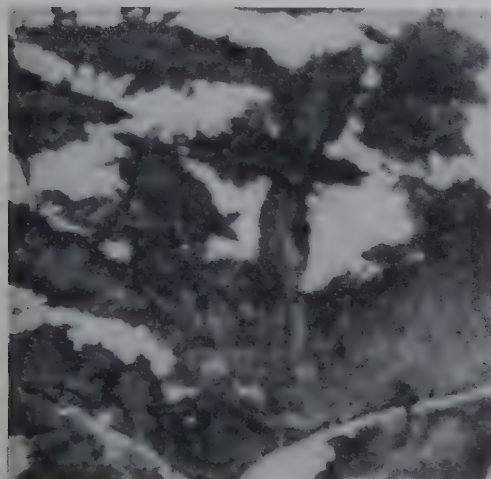
that previously found<sup>1</sup> in tempered martensite residues in which indications of individual particles were not observed. As time at temperature and degree of transformation increase, the structure becomes even more granular,

probably reflecting an increase in the size of the individual particles. It has also been observed that residues of bainitic carbon steels transformed in shorter periods are intermediate in appearance between the bainitic chro-

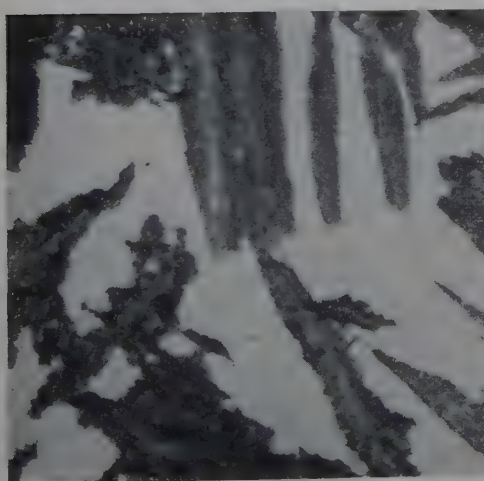




**FIG 16—Steel C—1.17 pct C, 5.95 pct Cr. Transformed 100 pct after 100 hr at 1300°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 17—Steel C—1.17 pct C, 5.95 pct Cr. Transformed 5 pct after 1 hr at 1000°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 18—Steel C—1.17 pct C, 5.95 pct Cr. Transformed 50 pct after 3 hr at 1000°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .

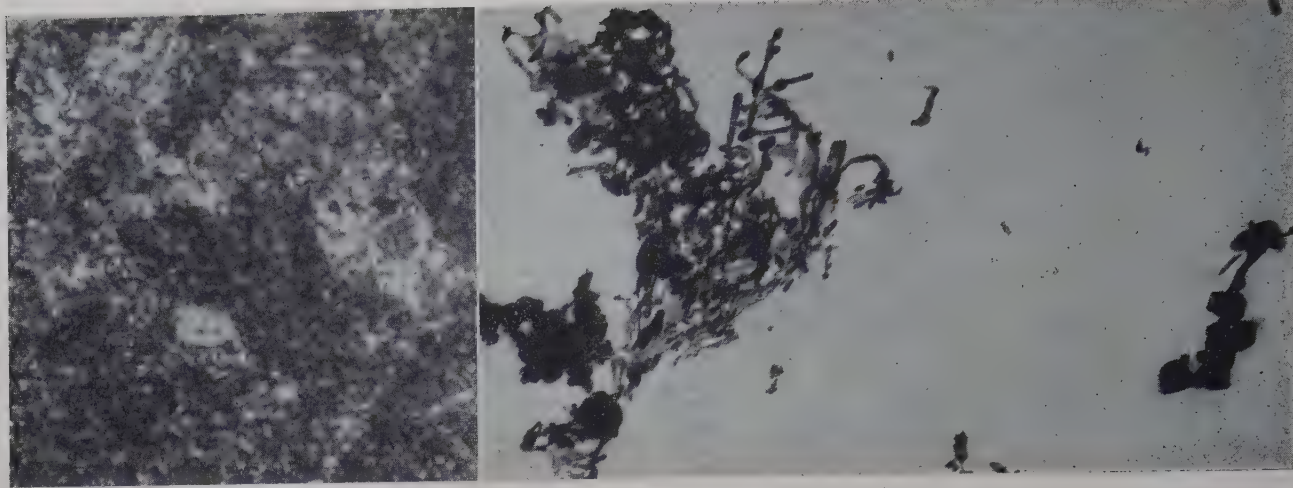
mium steel residues and martensitic residues, and it is inferred that the growth of the fine particles to dimensions visible at this magnification results from a tempering process.

In a similar manner the thickened

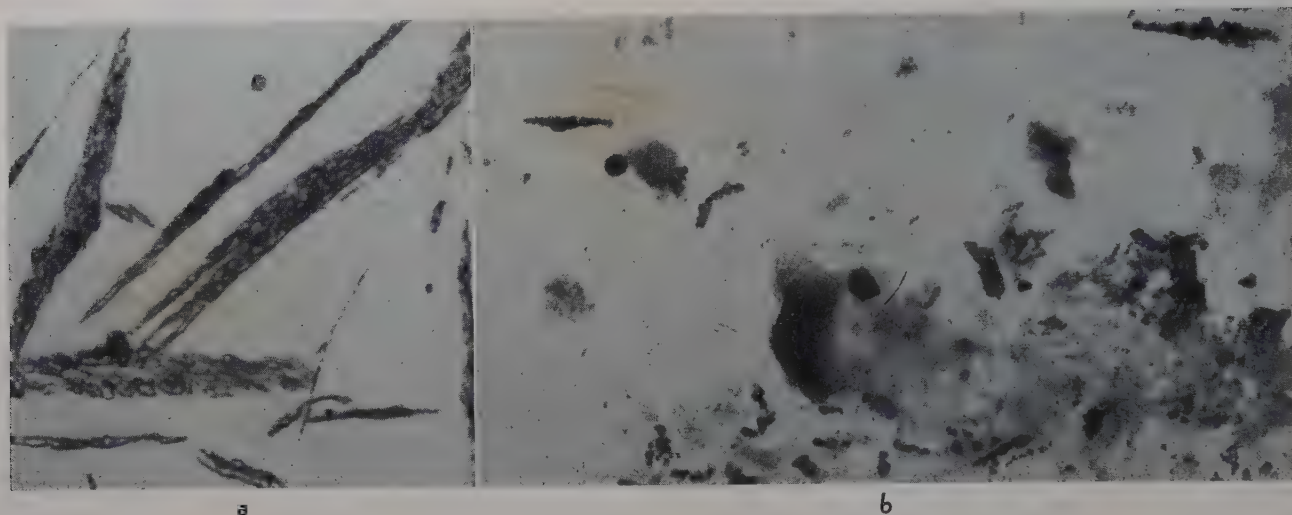
planes that are seen in the cloudy matrix are significantly different from similar planes in martensite residues. The character of these planes is best shown in Fig 13 where three small zones are separated from the main

cluster. These are believed to be the fragments of the thickened planes that appear to contain larger particles than the matrix. The size of the particles and possibly their number increase with time at temperature. Such structures





**FIG 19—Steel C—1.17 pct C, 5.95 pct Cr. Transformed 98 pct after 100 hr at 1000°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .



**FIG 20—Steel C—1.17 pct C, 5.95 pct Cr. Transformed 5 pct after 100 hr at 600°F.**  
A—Steel structure.  $\times 2000$ . B—Extracted residue. Electron micrograph.  $\times 25,000$ .

have not been observed in tempered martensite where the density of the matrix is lessened by precipitation along many planes in the form of continuous two-dimensional plates rather than a number of fine particles. The well defined larger particles, shown best in Fig 7 and 20, seem to be of a different character and may have been formed by a different mechanism than that by which the main structure was formed.

Although it is not presumed that these structures are adequate to answer the question of whether bainite forms initially as a single phase or as an aggregate, the structures are quite suggestive. With respect to these steels it is evident that, if it be assumed that the bainite formed initially by a martensite-like reaction and that it was tempered immediately, the resulting structure is significantly different from that formed when a truly martensitic structure is tempered by reheating.

Thus, if bainite in these steels were formed by the decomposition of a martensite-like constituent, either the transitional structure or the manner of its tempering is significantly different from martensite. However, the structure is so closely related to that of martensite that it is not possible to infer, as an obvious corollary, that bainite forms as an aggregate. An examination of bainite residues at much higher magnification would be desirable to obtain a better understanding of the structure.

### Summary

Examination of carbide residues extracted electrolytically from chromium steels has been shown by X ray diffraction to be consistent with the normally expected constituents of these steels, except for observation of two forms, differing in lattice dimensions, of  $\text{Cr}_7\text{C}_3$  and of an unidentified transi-

tional constituent in pearlitic specimens. Electron micrographs of the residues have shown characteristic forms of carbide for pearlite and bainite.

The carbides associated with pearlite are of two shapes. One is a thin irregularly shaped plate with thickened edges that is considered to be the lamellar carbide of regular pearlite and the other is a skeletal, rod-like or massive type that is considered to be the nonlamellar carbide in confused pearlite. The more extended forms of both types tend to coagulate locally, and the resulting massive particles, as well as the initially formed massive type carbides, grow by diffusion until they assume idiomorphic shapes in spheroidized pearlite structures. The tendency toward either type is controlled by the conditions of transformation rather than by specific effects of composition, and similar appearing carbides may be produced regardless of whether the carbide is of





FIG 21—Electron micrograph of shadow-cast residues of Steel C.  $\times 10,000$ .  
Transformed 65 pct after 3.25 min. at 1300°F.



FIG 22—Electron micrograph of shadow-cast residues of Steel C.  $\times 10,000$ .  
Transformed 100 pct after 100 hr at 1300°F.

the  $\text{Fe}_3\text{C}$  or  $\text{Cr}_7\text{C}_3$  type.

Residues from bainitic structures are closely analogous to those of martensite, but are significantly different. In bainite the residues have a nebulous, but granular, matrix, and discrete particles are found in the planes on which carbide tends to concentrate, rather than the unresolvable matrix and the continuous plates of tempered martensite residues. The bainite residues also contain fairly large particles not found in martensite residues. These characteristics suggest that although bainite may be formed in a manner analogous to martensite, the differences in structure are sufficiently great to indicate a significant difference in the mechanism. Further examination at much higher magnification should be helpful in understanding bainite formation.

Although this study of extracted carbides has been limited to the cursory examination of a few steels, it would appear that this use of the electron microscope is capable of adding considerably to our understanding of the structures of steel.

### Acknowledgments

The authors wish to acknowledge the generous cooperation of the staff of the Union Carbide and Carbon Research



FIG 23—Electron micrograph of shadow-cast residue of Steel B.  $\times 25,000$   
Transformed 95 pct after 100 hr at 600°F.

Laboratories, Inc. Special acknowledgment is due to W. D. Forgeng for the metallographic and X ray studies and to A. C. Jenkins of the Research Laboratories of The Linde Air Products Co. for preparing the electron micrographs.

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ary Hardening of Tempered Martensitic Alloy Steels. *Trans. AIME* (1949) 180. *Metals Tech.*, Sept. 1948, TP 2439.

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# The Properties of Sand Cast Magnesium-Rare Earth Alloys

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## Introduction

Several publications<sup>1-7</sup> during the past few years have demonstrated the markedly greater effect of cerium, as compared to all other alloying elements, in enhancing the strength and creep resistance of magnesium at elevated temperature either with or without the presence of other elements. In the work reported in Ref. 1-6, the cerium was added in the form of Mischmetal, which contains all the rare earth elements in essentially the same proportions as they occur in monazite sand, the principal ore of these metals. Mellor and Ridley<sup>7</sup> have presented the creep characteristics at 200°C of magnesium alloys prepared both with pure cerium and with Mischmetal. Up to the present time, however, no comprehensive study of the effect of the various component elements of Mischmetal on the properties of sand-cast magnesium has been made. This hiatus in magnesium technology undoubtedly is associated with the difficulty in obtaining the rare earth metals separately.

This investigation provides such a survey, the object of which was to determine which of the elements present in Mischmetal contributes the greatest effect in developing high strength and high resistance to creep at elevated temperatures. Toward this end the compositional variation of these properties has been determined as a function of temperature in the following alloy systems:

1. Magnesium-Mischmetal
2. Magnesium-cerium-free Mischmetal
3. Magnesium-didymium
4. Magnesium-praseodymium-lanthanum
5. Magnesium-cerium

Table 1 . . . Chemical Analyses of Rare Earth Metals

Producer's Designation of Material	Pct Total Rare Earths	Pct Ce	Pct Nd	Pct Pr	Pct Th†	Pct La*	Pct Fe	Pct Si	Pct Cr	Pct Other Impurities‡
Mischmetal . . . . .	97.8	50.6	18.2	6.4	nil	22.6	0.59	0.16	0.03	1.42
Ce-free Mischmetal . . . . .	94.5	0.2	35.2	12.2	0.9	46.0	3.24	0.20	0.31	1.79
Didymium . . . . .	89.8	0.8	72.3	7.9	nil	8.8	7.05	0.56	0.75	2.84
Cerium . . . . .	94.8	92.2	0.9	0.3	nil	1.4	2.37	0.02	0.50	2.21
Lanthanum . . . . .	96.5	0.5	0.7	0.2	nil	95.1	1.77	0.08	0.25	1.40
Praseodymium . . . . .	95.2	0.8	2.9	65.6	nil	25.9	2.61	0.60	0.19	1.40

\* Lanthanum obtained by difference from total rare earth content and therefore includes all the lesser elements such as Sa, Tb, Y, etc.  
† Thorium included in Total Rare Earth Content.  
‡ Other impurities obtained by difference from 100 pct. These impurities consist of oxygen and probably Al, Mn, Mg, and Cu in varying amounts. Some carbides and nitrides are also present.

Table 2 . . . Typical Analysis of Electrolytic Magnesium by Spectrographic Method

Al	Ca	Cu	Fe	Mn	Ni	Pb	Si	Sn	Zn
<0.01	<0.01	<0.01	0.03	0.05-0.10	<0.001	<0.001	<0.01	<0.001	<0.01

6. Magnesium-lanthanum  
This designation, based on the names of the metals as furnished by the producer, is used throughout the paper in order to avoid the complicated system of listing all the elements present in the more complex alloys. The composition of the metals and the alloys is discussed in detail below.

## Preparation of Alloys

### ALLOYING INGREDIENTS

The chemical composition of each of

the six alloying ingredients used to prepare the alloys reported in this paper is given in Table 1. In each case, the percent total rare earths, percent cerium, percent neodymium, percent praseodymium, and percent thorium were determined in addition to the listed impurities. The percent lanthanum was determined by difference from the total rare earth content and consequently includes the other elements (samarium, terbium, yttrium, etc.) present in Mischmetal. These additional elements, however, seldom amount to more than 1 to 2 pct of the total rare earth content of Mischmetal. The iron content of the Mischmetal is considerably lower than that of the other metals. This grade of Mischmetal is now readily available.

The metals are listed in the order of decreasing complexity. Elimination of the cerium from Mischmetal results in what the trade calls "Cerium-free Mischmetal." Carrying the separation a step further by eliminating the lantha-

New York Meeting, February 1950.  
TP 2726 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before April 1, 1950. Discussion is tentatively scheduled for publication in November, 1950.  
Manuscript received April 22, 1949; revision received Aug. 18, 1949.  
\* Metallurgist, Magnesium Laboratories, The Dow Chemical Co., Midland, Michigan.  
† References are at the end of the paper.

num yields what is commonly called "didymium" and should consist essentially of neodymium and praseodymium. Unfortunately, the material obtained for this work also contains 8 pct lanthanum (10 pct of the total rare earths) and the praseodymium content is rather low compared to that of Ce-free Mischmetal. The cerium and the lanthanum metals are both practically free of other rare earth metals. The praseodymium, however, contains a large proportion of lanthanum; consequently, the alloys made with it are called magnesium-praseodymium-lanthanum alloys.

MELTING AND CASTING OF ALLOYS

All the alloys used in this study were prepared in small laboratory melts according to the melting practice described by Nelson<sup>8</sup> as the "Crucible Method." The special precautions necessary in the alloying of Mischmetal with magnesium have been described in detail by Marande.<sup>9</sup> The same procedure was followed in introducing the other rare earth metals into magnesium.

A 50- to 100-lb melt of each alloy was prepared except for the didymium alloys and the praseodymium-lanthanum alloys, in which case smaller melts were necessitated by the smaller amounts of alloying ingredients available. Test bars of 6½ in. length with a 2½ in. long reduced section of ½ in. diam were cast in sand molds using a four-bar pattern. Electrolytic magnesium was used as the starting material; a typical analysis of this grade of magnesium is shown in Table 2.

In order to conserve the rare-earth metals, the alloy containing the highest percentage of the added element in each group was made first. Alloys of lower content were then prepared by remelting the scrap from the preceding melt and adding magnesium to obtain the desired composition of the next alloy in the series. All the melts were poured at 1300-1325°F in an attempt to obtain as fine a grain size as possible.

ANALYSIS

Chemical analysis to determine the rare earth content of each alloy was made on a sample taken from a test bar, whereas the spectrographic analysis of impurities was performed on separately cast specimens poured immediately before filling the test bar molds. The results of both analyses are given in Table 3; only the impurities which are present in major proportions

or those which differ markedly from the amounts present in electrolytic magnesium (Table 2) are tabulated.

As indicated in Table 3, two alloys from each series were analyzed for cerium, neodymium, praseodymium, and total rare earths; in all the other alloys, only the total rare earth content was determined. The lanthanum values represent the difference between cerium + neodymium + praseodymium and the total rare earths as mentioned

above. The method of analysis of cerium and of total rare earths is given in another publication.<sup>10</sup> These analyses are accurate to ±0.10 pct rare earth. The neodymium and the praseodymium were determined by the spectrophotometric method described by Rodden.<sup>11</sup> The accuracy of the analyses by this method is believed to be ±5 pct for neodymium and ±10 pct for praseodymium. The same analytical methods were used in determining the composi-

Table 3 . . . Composition and Grain Size of Alloys

Alloy Number	By Chemical Analysis					By Spectrographic Analysis					Grain Size 0.001 in.
	Pct Total Rare Earths	Pct Ce	Pct Nd	Pct Pr	Pct La*	Pct Al	Pct Fe	Pct Mn	Pct Pb	Pct Si	
Magnesium-Mischmetal Alloys											
1	0.18	0.09	0.03	0	0.06	<.01	0.041	0.17	0.005	<.01	14-28
2	0.40	0.21	0.06	0.02	0.11	<.01	0.06	0.18	0.008	<.01	14-16
3	1.15	0.59	0.19	0.06	0.31	0.014	0.030	0.14	0.023	0.018	60-90 (10-80 pct Columnar)
4	1.43	0.67	0.22	0.07	0.35	0.015	0.019	0.14	0.022	0.05	
5	1.62	0.83	0.27	0.09	0.43	0.026	0.042	0.16	0.014	<.01	45-60
6	2.50	1.28	0.42	0.14	0.66	<.01	0.009	0.17	0.007	0.02	35-60 (10-30 pct Columnar)
7	2.85	1.47	0.47	0.16	0.75	0.023	0.042	0.15	0.006	<.01	14-22
8†	3.23	1.69	0.51	0.17	0.86	<.01	0.048	0.09	0.005	<.01	20-28
9†	6.33	3.21	1.10	0.36	1.66	<.01	>.05	0.06	0.005	<.01	12-16
10	10.11	5.20	1.68	0.56	2.67	<.01	>.05	0.04	0.007		10-14
Magnesium-Ce-free Mischmetal Alloys											
11	0.14	0	0.05	0.01	0.08	<.01	0.035	0.11	0.031	0.02	70-100 pct Columnar
12	0.26	0	0.08	0.02	0.16	0.015	0.022	0.12	0.028	0.04	80-100 pct Columnar
13	0.51	0	0.17	0.04	0.30	0.015	0.021	0.10	0.025	0.04	50-70 (30-70 pct Columnar)
14	0.93	0.01	0.31	0.08	0.53	0.019	0.024	0.12	0.042	0.05	40-70 (10-60 pct Columnar)
15	1.87	0.03	0.62	0.17	1.05	0.034	0.057	0.08	0.013	<.01	40-50 (20-40 pct Columnar)
16	1.89	0.03	0.62	0.17	1.07	<.01	0.064	0.11	0.035	<.01	30-40
17	2.11	0.03	0.70	0.19	1.19	<.01	0.030	0.12	0.039	<.01	30-45
18	2.59	0.04	0.85	0.23	1.47	<.01	0.020	0.17	0.030	<.01	18-30
19†	3.00	0.07	0.86	0.21	1.86	<.01	0.044	0.12	0.015	<.01	12-16
20	5.05	0.07	1.67	0.45	2.86	<.01	0.052	0.16	0.047	<.01	8-15
21	5.74	0.03	2.14	0.62	2.94	0.039	0.051	0.10	0.020	<.01	12-14
Magnesium-didymium Alloys											
22	0.65	0.01	0.51	0.06	0.07	0.019	0.032	0.04	0.008	<.1	30-50 (30-80 pct Columnar)
23	1.63	0.02	1.29	0.14	0.18	<.01	0.040	0.07	0.010	<.01	40-80 (10-70 pct Columnar)
24	2.60	0.03	2.05	0.22	0.30	0.043	0.044	0.09	0.017	0.02	16-28
25†	3.85	0.06	3.00	0.31	0.48	<.01	0.045	0.09	0.020	<.01	12-20
26†	6.03	0.07	4.82	0.56	0.58	<.01	0.062	0.09	0.027	<.01	16-24
Magnesium-praseodymium-lanthanum Alloys											
27	0.69	0.0	0.02	0.47	0.20	<.01	0.030	0.07	0.004	<.01	40-50 (30-70 pct Columnar)
28	1.59	0.01	0.05	1.08	0.45	<.01	0.040	0.07	0.008	<.01	30-40
29†	3.00	0.02	0.11	2.03	0.84	<.01	0.033	0.08	0.011	<.01	14-20
30†	6.19	0.05	0.17	4.20	1.77	<.01	0.030	0.07	0.023	<.01	8-14
Magnesium-cerium Alloys											
31	0.15	0.15	0	0	0	<.01	0.030	0.14	0.013	0.02	80-100 pct Columnar
32	0.45	0.44	0	0	0.01	<.01	0.013	0.12	0.033	0.03	80-90 pct Columnar
33	1.09	1.05	0.01	0	0.03	<.01	0.038	0.14	0.030	0.02	30-60 (10-30 pct Columnar)
34	1.54	1.49	0.02	0.01	0.03	<.01	0.024	0.14	0.030	0.04	30-50 (20-40 pct Columnar)
35	2.01	1.95	0.02	0.01	0.03	<.01	0.019	0.18	0.030	<.01	60-80 (40-80 pct Columnar)
36	2.39	2.31	0.03	0.01	0.04	<.01	0.038	0.17	0.025	<.01	30-50
37†	2.60	2.48	0.04	0.02	0.06	<.01	0.037	0.14	0.018	<.01	30-40
38	3.57	3.45	0.04	0.02	0.06	<.01	0.040	0.12	0.031	<.01	10-20
39†	5.70	5.56	0.05	0.02	0.07	0.028	0.062	0.10	0.014	<.01	50-80
40	6.73	6.50	0.08	0.04	0.11	<.01	>.05	0.06	<.001	<.01	14-16
Magnesium-lanthanum Alloys											
41	0.20	0	0	0	0.20	<.01	0.014	0.11	0.028	0.04	28-38
42	0.32	0	0	0	0.32	<.01	0.028	0.10	0.030	0.04	40-60
43	0.54	0	0	0	0.54	<.01	0.004	0.14	0.024	0.03	40-60 (10-20 pct Columnar)
44	0.89	0.01	0	0	0.88	0.011	0.022	0.04	0.022	0.04	40-50 (10 pct Columnar)
45	1.39	0.01	0.01	0	1.37	<.01	0.003	0.10	<.01	<.01	35-100 (10-30 pct Columnar)
46	1.60	0.02	0.01	0	1.57	0.012	0.048	0.12	0.020	0.04	60-80 (20-70 pct Columnar)
47	2.47	0.02	0.01	0	2.44	<.01	0.044	0.11	0.011	<.01	40-50 (10 pct Columnar)
48	2.74	0.03	0.02	0	2.69	<.01	0.016	0.10	0.022	<.01	20-30
49	2.80	0.03	0.02	0	2.75	<.01	0.030	0.12	0.006	0.02	60-70
50	3.59	0.04	0.02	0.01	3.52	<.01	0.045	0.14	0.008	0.04	60-100 (10-50 pct Columnar)
51†	3.85	0.04	0.02	0.01	3.78	0.014	0.032	0.08	0.022	0.02	30-35
52	6.51	0.06	0.04	0.02	6.39	<.01	0.045	0.09	0.006	<.01	30-80
53†	6.70	0.06	0.04	0.02	6.58	0.03	0.041	0.12	0.047	0.02	10-18

\* By difference from Total Rare Earth Content.  
† Alloys analyzed separately for Ce, Nd, Pr, and Total Rare Earths.



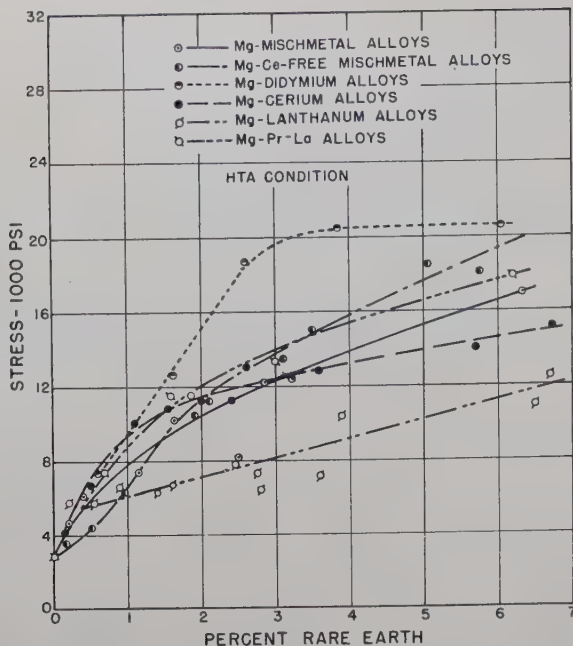


FIG 1—Tensile yield strength at room temperature.

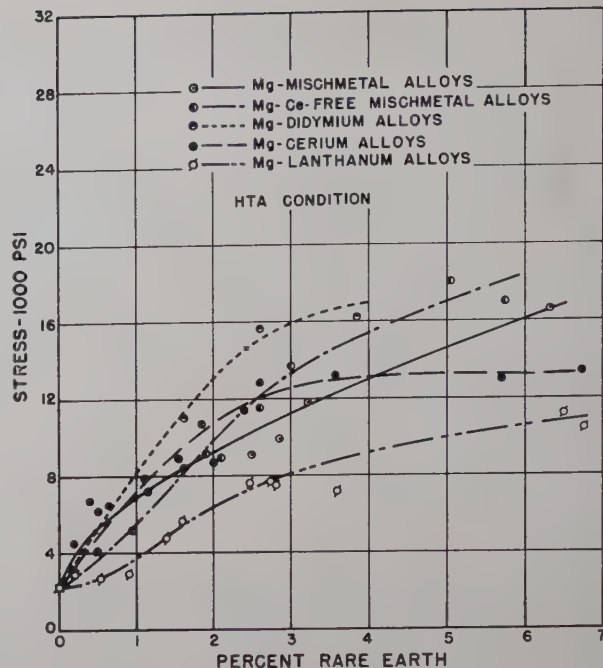


FIG 2—Tensile yield strength at 200°F.

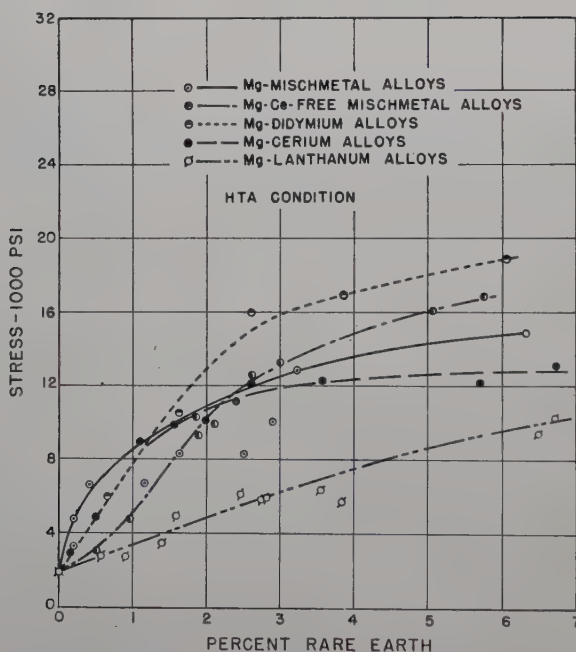


FIG 3—Tensile yield strength at 300°F.

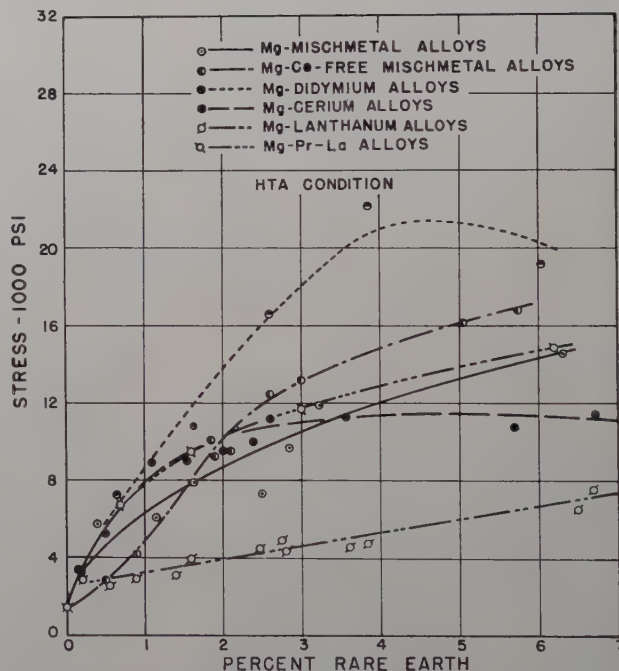


FIG 4—Tensile yield strength at 400°F.

tion of the rare earth metals given in Table 1.

In the alloys analyzed for the separate rare earth metals, the proportion of each of the four elements agrees reasonably well with that of the same element in the corresponding alloying ingredient. This observation, together with the good agreement between the two alloys analyzed in each group, indicates that there is no preferential loss of any of these elements either in alloying with magnesium or in remelting. With these findings on hand,

it was deemed justifiable to apportion the total rare earth content of all the other alloys among the four elements. For each series of alloys, the proportioning factors used were based on the average of the two complete analytical determinations in the series.

#### HEAT TREATMENT

A preliminary study was conducted to determine the maximum possible solution heat treating temperature for each type of alloy. This was done by

heat treating small specimens of alloys containing 6 pct and 3 pct rare earths in the as-cast condition at a series of temperatures followed by quenching in cold water. Metallographic examination of these specimens has established the following minimum temperatures of liquation:

	°F
Magnesium-Mischmetal alloys.....	1095 ± 5
Magnesium-Ce-free Mischmetal alloys.....	1100 ± 5
Magnesium didymium.....	1000 ± 5
Magnesium-praseodymium-lanthanum alloys.....	1100 ± 5
Magnesium-cerium alloys.....	1095 ± 5
Magnesium-lanthanum alloys.....	1125 ± 5

These temperatures are in good agree-

Table 4 . . . Tensile Properties of Alloys in the As-cast, Heat Treated, and Heat Treated + Aged Conditions at Room Temperature

Alloy Number	Pct Total Rare Earths	As-Cast			Heat Treated			Heat Treated + Aged		
		Pct E	TYS	TS	Pct E	TYS	TS	Pct E	TYS	TS
Magnesium		6.0	2.9	12.6						
Magnesium-Mischmetal Alloys										
1	0.18	9.5	4.4	20.4	10.2	4.2	20.9	10.0	4.7	21.4
2	0.40	9.1	5.1	20.7	7.8	4.0	13.8	8.2	6.1	16.6
3	1.15	4.6	5.3	14.3	6.0	5.3	16.0	6.0	7.4	18.0
5	1.62	2.9	7.0	14.4	5.5	7.8	20.0	3.5	10.2	19.3
6	2.50	1.9	8.7	15.5	3.0	6.8	15.4	2.2	8.2	17.0
7	2.85	1.7	9.0	15.4	5.0	10.5	18.6	1.7	12.2	19.4
8	3.23	1.1	9.1	14.9	3.4	8.9	18.7	1.5	12.4	20.5
9	6.33	0.5	13.1	16.6	2.0	13.8	16.8	1.0	17.0	20.4
Magnesium-Ce-free Mischmetal Alloys										
11	0.14	6.6	3.0	14.3	6.5	3.2	15.3	6.2	3.6	14.1
12	0.26	5.3	3.5	12.2	5.2	3.9	12.4	5.5	4.2	13.5
13	0.51	5.7	4.0	14.1	6.5	4.0	15.3	6.0	4.4	15.0
14	0.93	4.8	5.0	14.0	5.7	6.3	15.5	5.7	6.4	16.0
15	1.87	3.5	6.9	15.9	5.2	6.7	16.1	3.5	11.5	19.4
16	1.89	4.3	6.9	16.3	7.5	7.7	20.1	3.2	10.4	21.5
17	2.11	4.1	7.5	16.4	5.8	8.5	19.5	2.3	11.2	21.3
18	2.59	3.5	8.6	16.6	6.0	8.8	20.5	2.2	13.5	23.0
19	3.00	3.1	9.4	17.3	7.0	10.1	20.2	2.2	15.0	23.4
20	5.05	0.5	12.7	16.0	2.7	12.0	19.3	0.7	18.5	24.5
21	5.74	1.3	13.1	17.3	4.0	13.1	20.6	1.5	18.1	25.4
Magnesium-didymium Alloys										
22	0.65	7.2	5.1	19.1	8.2	4.5	19.4	5.7	7.3	22.3
23	1.63	6.7	7.2	19.5	7.2	7.2	16.8	5.0	12.6	23.4
							21.6			29.0
24	2.60	5.0	11.3	21.0	9.0	11.8	25.2	3.7	18.7	31.9
25	3.85	3.0	13.8	21.9	4.2	14.1	23.7	2.2	20.5	28.9
26	6.03	0.5	18.5	20.7	1.5	14.7	20.2	0.5	20.6	25.7
Magnesium-cerium Alloys										
31	0.15	5.2	3.3	12.6	6.0	4.0	11.9	4.5	4.2	14.2
32	0.45	5.0	4.2	13.7	5.0	5.0	16.4	5.0	6.7	20.2
33	1.09	5.5	5.6	16.1	3.5	6.9	18.9	2.5	10.0	22.3
34	1.54	4.1	6.3	15.2	6.0	8.8	21.5	3.7	10.8	21.6
35	2.01	2.5	6.6	12.8	6.3	8.9	18.0	2.3	11.2	18.4
36	2.39	1.9	8.3	14.5	3.7	7.4	16.7	1.7	11.2	18.2
37	2.60	1.9	8.9	15.3	3.7	9.8	18.4	1.5	13.0	19.9
38	3.57	1.0	9.9	14.9	4.2	10.6	17.4	1.7	12.8	19.1
39	5.70	0.5	12.5	14.0	1.0	12.2	14.4	1.5	14.0	15.6
40	6.73	0.7	17.1	19.5	1.2	12.1	16.6	0.5	15.2	18.0
Magnesium-lanthanum Alloys										
41	0.20	6.6	4.5	17.1	6.7	5.7	18.0	7.0	5.8	18.3
43	0.54	5.3	4.8	15.6	5.7	5.2	17.0	6.0	5.8	16.9
44	0.89	4.5	5.5	15.1	5.2	6.0	17.0	4.5	6.6	17.1
45	1.39	2.6	5.4	10.2	4.0	6.5	11.6	3.5	6.3	12.0
46	1.60	3.2	5.9	13.0	3.5	6.5	15.0	4.0	6.7	15.0
47	2.47	2.0	8.1	14.2	3.2	7.8	15.4	3.2	8.1	15.3
48	2.74	1.5	8.4	14.8	2.5	7.1	14.5	3.0	7.4	15.2
49	2.80	1.1	7.8	11.2	2.7	6.3	12.3	2.7	6.4	12.1
50	3.59	0.6	8.7	11.2	1.7	6.9	11.0	1.7	7.2	11.8
51	3.85	1.0	9.9	15.1	2.0	10.0	15.0	1.7	10.4	14.9
52	6.51	0.0	14.1	15.0	0.5	10.7	14.4	1.0	11.0	14.8
53	6.70	0.0	14.1	17.7	0.5	12.0	15.6	1.0	12.5	16.1

Pct E—Percent elongation in 2 in.  
TYS—Tensile yield strength in 1000 psi  
TS—Tensile strength in 1000 psi

ment with the eutectic temperatures of the published magnesium-rare earth diagrams;<sup>12,13,14,15</sup> no information is available on the principal constituent of didymium, that is, the magnesium-neodymium system.

With these determinations as a guide, the following heat treating temperatures were selected and used throughout this investigation:

Magnesium-didymium alloys.....	975
All other alloys.....	1070

The higher temperature possible with magnesium-lanthanum alloys was not used in order to avoid the dangers of oxidation and sagging of the test bars. All heat treatments were of 24-hr duration followed by cooling on a thick iron

plate under a fan blast. The aging treatment consisted of heating for 16 hr at 400°F.

All the heat treatments were conducted in circulating air furnaces, electrically heated and controlled to  $\pm 5^\circ\text{F}$ . The atmosphere of the furnace was rendered protective by the maintenance of a concentration of 0.5 to 1.0 pct sulphur dioxide.

GRAIN SIZE

The grain size of each alloy (Table 3) was determined on a sample from the reduced section of every test bar after being tested either in tension or in creep. The measurements were made

by the comparison method described by P. F. George.<sup>16</sup> A range of grain size is given for each alloy. This represents the variation from bar to bar. The figures given for columnar structure represent the percent of the area covered by columnar grains. It may be stated that, in general, the grain size decreases with increasing alloy content. The suppression of a columnar structure in the lower alloy contents is very erratic. No evidence of grain coarsening upon heat treatment was observed.

Testing Methods

Detailed descriptions of the methods of tension and creep testing have been given in earlier publications.<sup>4,17</sup> All the properties were determined on cast test bars without machining the surface. Tensile properties are the average of two tests. All creep data are from tests of 100 to 160 hr duration. Space does not permit the presentation of detailed creep data. The creep characteristics of each alloy are defined in this paper by three parameters:

- 1. Limiting stress to give 0.1 pct creep extension in 100 hr
- 2. Limiting stress to give 0.2 pct total extension in 100 hr
- 3. Limiting stress to give 0.5 pct total extension in 100 hr.

These values were determined by interpolation of log-log plots of stress vs. extension as described previously.<sup>4,17</sup> A minimum of 3 and as many as 8 stress levels were used to establish the log stress vs. log extension relationship for each alloy at each temperature. The total extension figures include the elongation obtained during loading as well as the creep extension. No significance has been attached to creep rates in this investigation because during the 100-hr test the secondary stage of creep, which gives the minimum creep rate, is not established in many tests.

The method of determining electrical conductivity has been described earlier.<sup>18</sup>

The density of each alloy in the heat treated plus aged condition was determined on a 5/8 in. diam by 1 in. long specimen machined from the shoulder of a test bar. Each sample was weighed on an analytical balance under two conditions: (1) in air, and (2) in kerosene of known density. The density of the alloy was calculated from these two weighings.

Discussion of Results

TENSILE PROPERTIES



The room temperature tensile properties of all the alloys in the as-cast, solution heat treated (HT), and heat treated and aged (HTA) conditions are presented in Table 4. The yield and tensile strengths in the HTA condition are plotted as a function of composition for all temperatures investigated in Fig 1 through 13. The total rare earth content of all the alloys is used to represent the composition in these as well as all subsequent plots. Graphs of elongation vs. composition have been omitted in order to conserve space; furthermore, the scatter in these data renders them rather inconvenient for graphical representation. Elongation values at all temperatures are given in Table 5.

Marked differences exist in the degree to which the strength of magnesium is enhanced by the various rare earth elements. The highest properties at all temperatures over the entire composition range investigated are developed by the addition of didymium, whereas the lowest properties are found in magnesium-lanthanum alloys. Intermediate to these lie the alloys containing cerium-free Mischmetal, praseodymium + lanthanum, Mischmetal, and cerium in that order of decreasing properties for alloys containing 3 to 6 pct total rare earths. Among these four types of alloys there is little difference in strength in compositions of less than 2 to 2.5 pct alloying elements. In general, the elongation decreases with increasing amounts of rare earth elements; the slight increase in this property in alloys containing 0.18 and 0.40 pct Mischmetal is undoubtedly associated with the unusually fine grain size of these alloys as compared to that of electrolytic magnesium which is 100 pct columnar.

It can readily be seen from the data in Table 4 that all the alloys, with the exception of the magnesium-lanthanum alloys, exhibit a certain degree of age hardening. The alloys may be rated in the following order of decreasing age hardening: magnesium-didymium, magnesium-cerium-free Mischmetal, magnesium-Mischmetal, magnesium-cerium, magnesium-lanthanum. From these observations it may be concluded that of the four principal elements in Mischmetal, neodymium contributes the most to age hardening. Quenching from the heat treating temperature into water at 180°F instead of air cooling effects no increase in the properties or in the degree of age hardening of any of these alloys. The

**Table 5 . . . Elongation of Alloys in the Heat Treated + Aged Condition at Room and Elevated Temperatures**

Alloy Number	Percent Total Rare Earths	Percent Elongation in 2 In.						
		Room Temp.	200°F	300°F	400°F	500°F	600°F	700°F
Magnesium		6.0	16.1	23.2	38.0	59.0	81.0	101.0
Magnesium-Mischmetal Alloys								
1	0.18	10.0	13.2	24.7	25.2	28.0	59.0	
2	0.40	8.2	20.5	24.0	23.7	28.5	52.0	107.6
3	1.15	6.0	10.7	13.2	18.2	18.7	29.5	65.0
5	1.62	3.5	11.0	15.0	15.0	19.0	32.5	76.6
6	2.50	2.2	3.0	5.8	8.5	16.5	32.7	56.6
7	2.85	1.7	5.0	10.0	9.7	11.2	28.2	
8	3.23	1.5	2.7	7.0	4.7	11.7	24.2	113.6
9	6.33	1.0	2.2	2.0	2.0	4.2	12.5	77.9
Magnesium-Ce-free Mischmetal Alloys								
13	0.51	6.0	12.0	17.5	20.0	20.7	25.5	70.7
14	0.93	5.7	12.2	15.5	19.0	16.3	25.5	58.3
15	1.87	3.5	5.2	8.5	11.0	14.5	22.2	
16	1.89	3.2	8.5	15.5	17.0	21.2	34.2	121.0
17	2.11	2.3	5.5	14.5	14.5	17.7	26.0	133.0
18	2.59	2.2	3.5	12.5	15.0	18.0	35.0	133.9
19	3.00	2.2	2.5	9.2	14.0	15.2	40.7	153.3
20	5.05	3.5	5.2	8.5	11.0	14.5	22.2	
21	5.74	1.5	2.5		4.5	9.2	34.0	146.0
Magnesium-didymium Alloys								
22	0.65	5.7	13.7	16.7	19.0	23.5	34.0	70.2
23	1.63	5.0	10.2	15.5	15.0	13.5	33.5	41.7
24	2.60	3.7	9.0	13.0	12.7	12.7	31.5	77.0
25	3.85	2.2	5.0	6.5	9.0	11.7	37.5	78.0
26	6.03	0.5		2.5	3.2	6.5	26.6	51.0
Magnesium-praseodymium-lanthanum Alloys								
27	0.69	4.5			18.0		39.0	
28	1.59	5.2			20.0		31.0	
29	3.00	2.7			14.5		38.0	
30	6.19	0.7			3.0		27.0	
Magnesium-cerium Alloys								
31	0.15	4.5	8.2	15.7	15.7	19.2	27.5	49.9
32	0.45	5.0	9.2	12.2	17.0	16.2	32.0	55.6
33	1.09	2.5	15.0	18.5	15.5	17.2	32.2	52.7
34	1.54	3.7	7.7	10.2	16.7	14.0	29.7	
35	2.01	2.3	4.2	9.7	13.0	16.5	27.2	55.7
36	2.39	1.7	3.2	9.0	14.5	18.7	38.5	78.3
37	2.60	1.5	4.7		11.2	17.2	31.5	83.6
38	3.57	1.7	3.7	7.0	13.5	12.0	40.0	109.3
39	5.70	1.5	2.0	2.0	3.2	6.5	32.5	90.6
40	6.73	0.5	1.2	1.7	3.0	3.2	33.0	62.3
Magnesium-lanthanum Alloys								
41	0.20	7.0	14.0	19.5	26.0	26.2	43.0	
43	0.54	6.0	13.0	19.2	24.0	22.5	36.5	
44	0.89	4.5	12.2	16.7	20.7	23.0	34.5	
45	1.39	3.5	7.0	10.5	24.0	23.0	24.2	
46	1.60	4.0	9.0	14.7	22.0	23.5	40.7	
47	2.47	3.2	7.5	13.7	23.0	21.0	45.0	
48	2.74	3.0	4.5	12.2	21.0	26.7	48.7	
49	2.80	2.7	4.2	9.5	19.2	22.0	28.2	
50	3.59	1.7	2.7	5.0	14.7	15.8	38.5	
51	3.85	1.7		9.0	18.5	23.5	48.7	
52	6.51	1.0	2.2	3.0	8.7	11.5	34.7	
53	6.70	1.0	1.5	2.5	7.5	10.5	40.0	

properties of magnesium-lanthanum alloys after heat treating at 1100°F are the same as those reported here after the 1070°F heat treatment.

The specific effect of each rare earth metal is more clearly illustrated by the property vs. temperature plots shown in Fig 14, 15, 16 and 17. These graphs have been derived from the property-composition curves for two levels of alloy content, 3 and 6 pct total rare earths; below 3 pct alloy content the properties decrease quite rapidly to the values for electrolytic magnesium. The curves in these figures do not necessarily pass through all the points read

from the property-composition plots, but deviations from them are no greater than 1000 psi.

#### EFFECT OF EXPOSURE AT 600°F

Table 6 shows the tensile properties of two compositions of magnesium-Mischmetal, magnesium-cerium-free Mischmetal, magnesium-didymium, magnesium-cerium, and magnesium-lanthanum alloys at room temperature and 600°F after holding the test bars at 600°F for 5000 hr. This long-time exposure treatment was conducted in an atmosphere containing  $\frac{1}{2}$  to 1 pct sulphur dioxide in order to prevent any

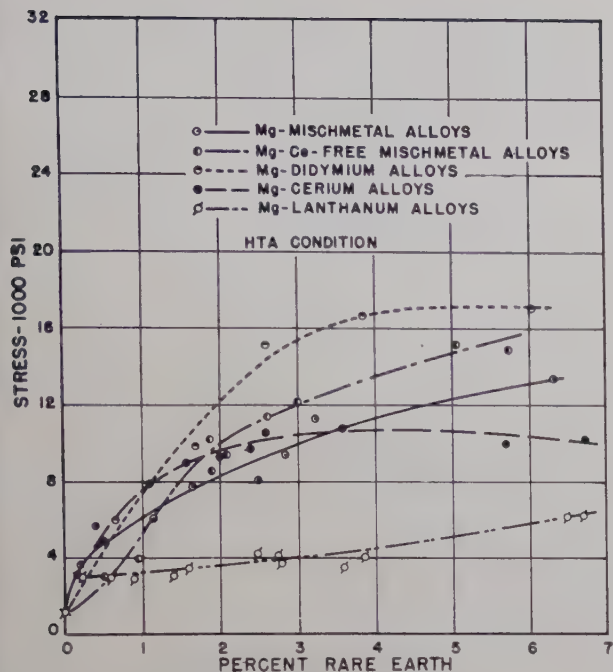


FIG 5—Tensile yield strength at 500°F.

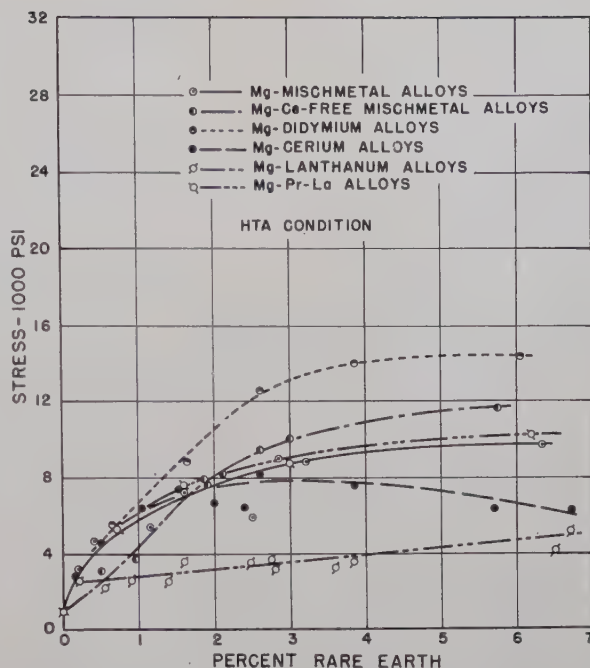


FIG 6—Tensile yield strength at 600°F.

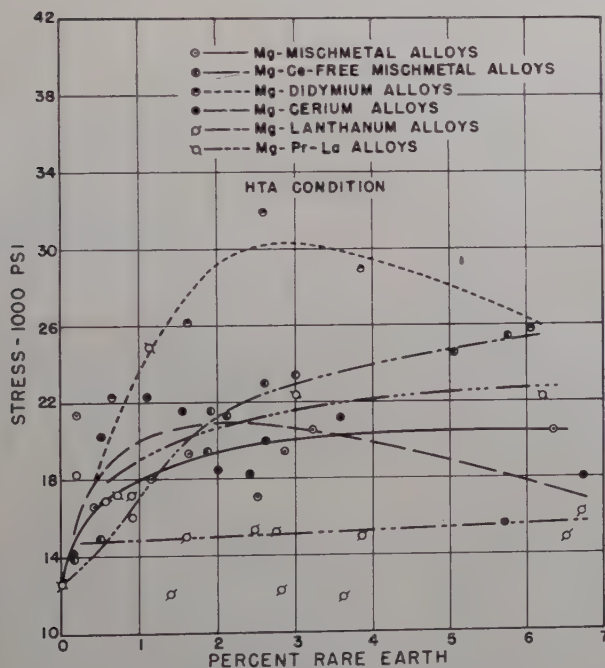


FIG 7—Tensile strength at room temperature.

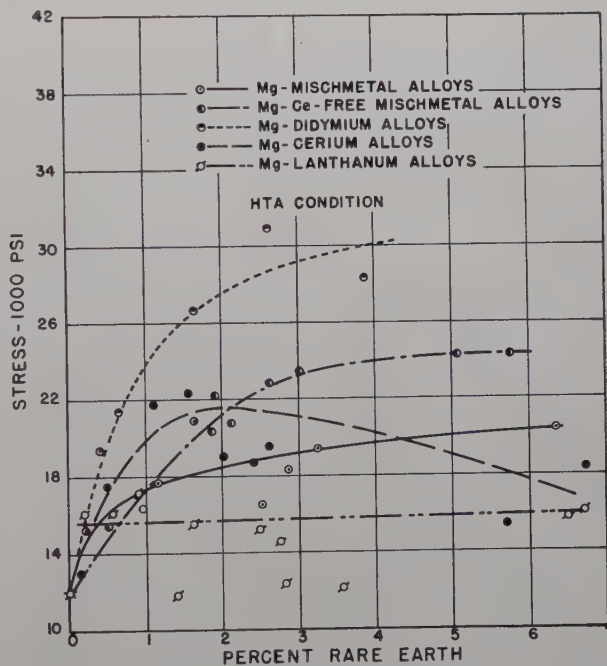


FIG 8—Tensile strength at 200°F.

preferential oxidation of the rare earth intermetallic compounds.

Compared to their properties in the HTA condition, the alloys containing Mischmetal, cerium-free Mischmetal, didymium, and cerium suffer a much greater loss in strength at room temperature and 600°F from this exposure than do the magnesium-lanthanum alloys. In fact, there is no change in the room temperature properties of magnesium-lanthanum alloys. However, the best combination of strength

at room temperature and 600°F after the prolonged exposure is exhibited by the magnesium + 5.74 pct cerium-free Mischmetal alloy.

Similar data have been reported by McDonald<sup>8</sup> for an alloy containing 6 pct Mischmetal plus 1.5–2.0 pct manganese. His results showed that the strength of this alloy decreases within 16 to 24 hr exposure at 600°F to a value that is maintained out to 1000 hr exposure. Subsequent experiments on the same material have been extended

to 5000 hr without any further change in the properties.

#### CREEP RESISTANCE

The creep characteristics of all six alloy systems at 400 and 600°F are depicted as a function of composition in Fig 18 through 21. Only a limited number of alloys, other than magnesium-Mischmetal alloys, have been investigated at 500°F; the results of these tests together with the data at



300°F for magnesium-Mischmetal alloys are summarized in Table 7.

The pronounced differences among the various rare earth metals in their respective effect on the creep resistance of magnesium at 400°F is demonstrated clearly by all three indices of creep resistance used to represent the data. All the rare earth elements, with the exception of lanthanum, effect a marked increase in this property in the composition range between 0 and 2 pct. In general, the alloys may be listed in the same order with respect to creep resistance as was found in their relative tensile properties. This listing applies specifically in the range of 3 to 6 pct rare earth content; in lower compositions some of the curves cross one another. The decrease in creep strength in magnesium-cerium alloys between 3 and 7 pct cerium content is difficult to explain. The reason does not lie in grain size variation inasmuch as of the two alloys containing 5.70 and 6.73 pct cerium, the former has a grain size of 0.050-0.080 in. and the latter 0.014-0.16 in. Furthermore, alloys of lower cerium content having higher creep resistance than either of the two high cerium compositions have grain size values which fall at both ends of the range covered by the high cerium alloys.

The results of tests at 500°F show much the same trends as indicated at 400°F. It will be noted that again there is a drop in creep resistance between 3 and 6 pct cerium. Alloys containing 1.15, 1.62, 2.50 and 2.85 pct Mischmetal exhibit anomalously low creep resistance at 300°F and high creep resistance at 500°F. These effects appear to correlate with the abnormally large grain size of these alloys.

The situation at 600°F is a little more confused; many anomalies exist in the data rendering them difficult to evaluate on a comparative basis. To illustrate these effects, Table 7a has been prepared from Fig 21 for three levels of alloy content:

These results may be summarized by saying that the relative merits of the various magnesium-rare earth alloys depend upon the temperature. Furthermore, there definitely appears to be a grain size effect, but it is not the same for all alloy systems. This effect is most pronounced in magnesium-cerium-free Mischmetal and magnesium-didymium alloys at 600°F, and in magnesium-Mischmetal alloys at 300 and 500°F. The selection of an alloy for applications demanding high creep resistance at elevated temperatures must, there-

Table 6 . . . Effect of 5000 Hr Exposure at 600°F on the Tensile Properties of Magnesium-rare Earth Alloys

Alloy Number	Pct Total Rare Earths	Tested at Room Temperature						Tested at 600°F					
		HTA			HTA + Exposure			HTA			HTA + Exposure		
		Pct E	TYS	TS	Pct E	TYS	TS	Pct E	TYS	TS	Pct E	TYS	TS
Magnesium-Mischmetal Alloys													
7	2.85	1.7	12.2	19.4	3.0	8.2	16.4	28.2	9.0	14.2	48.0	3.5	8.2
9	6.33	1.0	17.0	20.4	1.0	10.3	14.1	12.5	9.8	14.3	34.2	4.4	9.8
Magnesium-Ce-free Mischmetal Alloys													
18	2.59	2.2	13.5	23.0	3.0	8.5	16.7	35.0	9.5	14.7	63.5	4.1	9.6
21	5.74	1.5	18.1	25.4	1.5	11.5	17.7	34.0	11.7	17.0	58.5	5.9	12.4
Magnesium-didymium Alloys													
24	2.60	3.7	18.7	31.9	7.0	3.8	21.4	31.5	12.6	15.9	63.5	4.4	10.4
26	6.03	0.5	20.6	25.7	3.0	9.5	15.7	26.2	14.4	19.3	46.0	5.2	13.2
Magnesium-cerium Alloys													
37	2.60	1.5	13.0	19.9	2.2	6.9	14.8	31.5	8.1	11.3	49.7	2.5	7.0
39	5.70	1.5	14.0	15.6	1.5	9.9	14.4	32.5	6.4	10.6	25.7	3.6	8.8
Magnesium-lanthanum Alloys													
48	2.74	3.0	7.4	15.2	4.0	7.4	15.4	48.7	3.7	7.4	75.5	2.4	5.2
53	6.70	1.0	12.5	16.1	1.0	12.8	15.5	40.0	5.2	10.8	44.5	3.6	8.2

Pct E—Percent elongation in 2 in.  
TYS—Tensile yield strength in 1000 psi  
TS—Tensile strength in 1000 psi

fore, take into consideration the temperature range as well as the stress level. In order to illustrate the range in properties that exists in magnesium alloys, the tensile properties and creep limits of three rare earth alloys are compared with those of two commercial casting alloys and magnesium-6 pct zinc-zirconium alloy<sup>18</sup> in Table 8. The superiority of the alloys containing rare earth metals over the other alloys at 400°F is clearly demonstrated. In addition, this table illustrates in a concise form the quantitative differences among the rare earth alloys.

ELECTRICAL CONDUCTIVITY

The results of electrical conductivity measurements are given in Fig 22 for various conditions of heat treatment. The experimental points have been omitted from these plots in order to render the curves more clearly discernible. It can be said that the curves

Table 7 . . . Creep Limits at 500°F

Alloy No.	Composition	Creep Limits— 1000 psi		
		0.1 pct Creep Extension	0.2 pct Total Extension	0.5 pct Total Extension
300°F				
2	Pct			
3	0.40 Mischmetal	5.5	5.2	5.7
5	1.15 Mischmetal	7.9	6.1	7.2
6	1.62 Mischmetal	9.0	6.8	8.7
7	2.50 Mischmetal	9.7	7.3	9.2
8	2.85 Mischmetal	10.1	7.3	9.0
9	3.23 Mischmetal	10.0	8.4	10.2
	6.33 Mischmetal	10.6	9.6	11.8
500°F				
2	0.40 Mischmetal	3.1	3.2	4.2
3	1.15 Mischmetal	5.6	5.0	6.1
5	1.62 Mischmetal	6.1	5.8	6.3
6	2.50 Mischmetal	6.5	5.5	7.4
7	2.85 Mischmetal	7.4	6.8	8.3
8	3.23 Mischmetal	4.7	4.7	6.2
9	6.33 Mischmetal	5.3	5.5	6.7
19	3.00 Ce-free Mischmetal	5.5	5.5	7.0
21	5.74 Ce-free Mischmetal	5.2	5.6	7.4
25	3.85 Didymium	6.0	5.9	7.8
38	3.57 Cerium	4.8	4.8	5.6
39	5.70 Cerium	3.7	3.8	4.5

Table 7a . . . Comparison of Creep Limits at 600°F

Rare Earth Metal	Creep Limit—1000 psi								
	1.5 pct Total Rare Earths			3 pct Total Rare Earths			6 pct Total Rare Earths		
	0.1 pct Creep	0.2 pct Total	0.5 pct Total	0.1 pct Creep	0.2 pct Total	0.5 pct Total	0.1 pct Creep	0.2 pct Total	0.5 pct Total
Mischmetal	1.9	2.1	2.7	2.3	2.5	2.9	2.5	2.7	3.1
Cerium-free Mischmetal	1.8	1.9	2.5	1.5	1.9	2.2	1.5	1.9	2.2
Didymium	1.7	2.1	3.2	1.0	1.4	1.7	1.0	1.4	2.6
Praseodymium + lanthanum	2.0	2.1	2.5	1.7	2.1	2.2	1.7	2.0	2.2
Cerium	2.3	2.5	2.7	2.4	2.5	2.9	1.8	2.1	2.4
Lanthanum	1.1	1.2	1.4	1.2	1.3	1.5	1.4	1.6	2.1

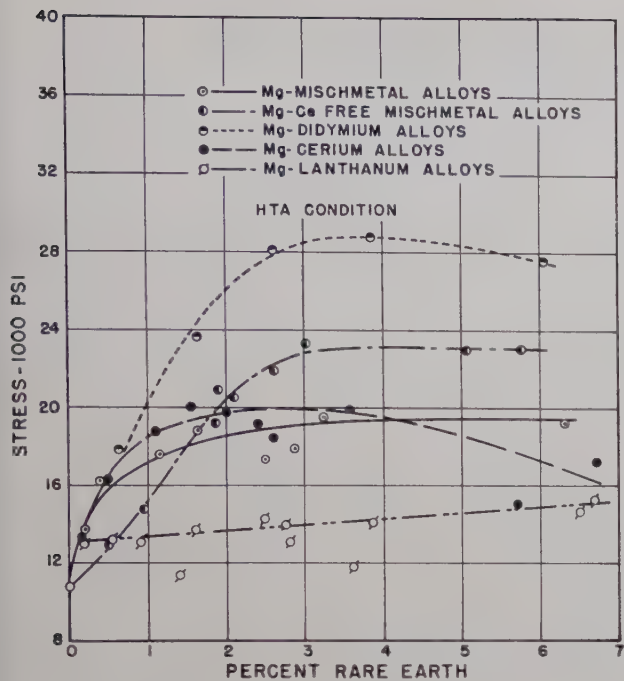


FIG 9—Tensile strength at 300°F.

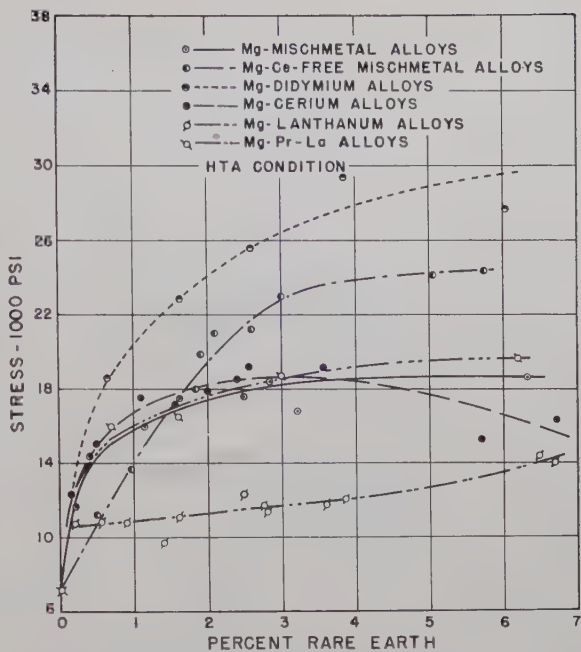


FIG 10—Tensile strength at 400°F.

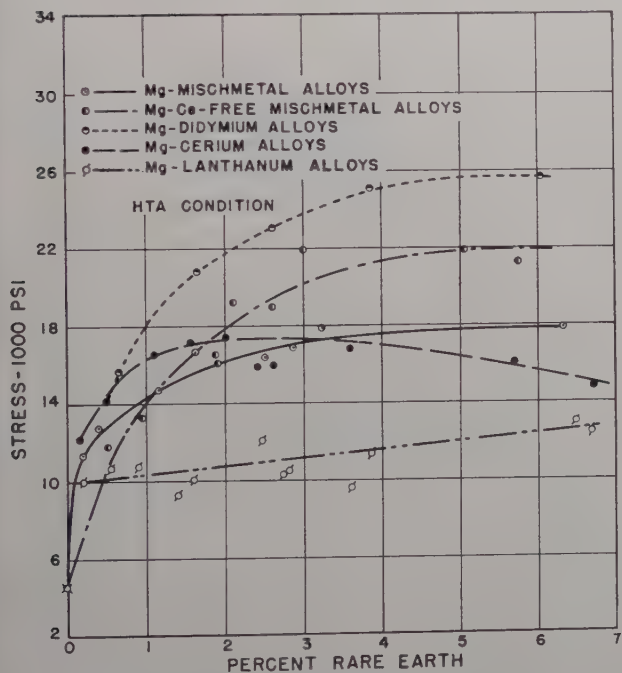


FIG 11—Tensile strength at 500°F.

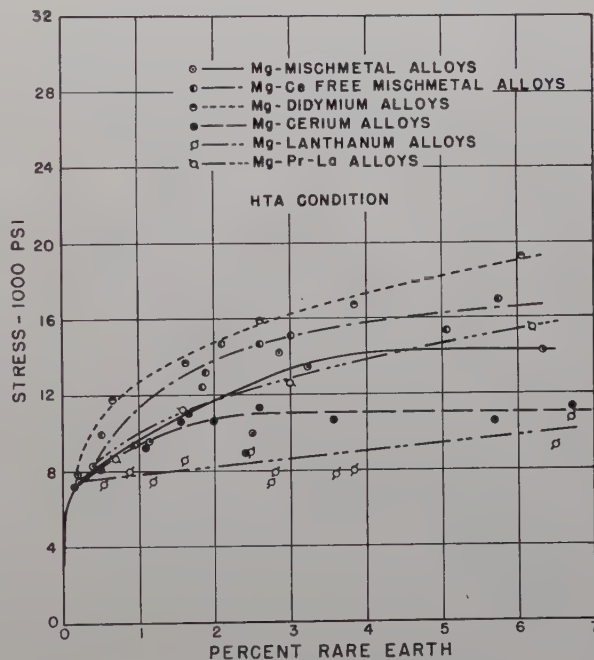


FIG 12—Tensile strength at 600°F.

as drawn follow faithfully the experimental data. The zero point of each series of curves is the conductivity of cast electrolytic magnesium in the same condition of heat treatment as the alloys in the series. The conductivity of this grade of magnesium is naturally a little lower than the value of 0.23 reciprocal microhm-cm reported for pure magnesium.

The decrease in conductivity with composition in each of the alloy sys-

tems is well illustrated by the curves. Considering the conductivity changes that take place upon aging, it may be predicted that the solid solubility of neodymium in magnesium is greater than that of any of the other rare earth elements. On the other hand, the relative insensitivity of the conductivity of magnesium-lanthanum alloys to heat treatment and to aging leads one to the conclusion that the solubility of lanthanum in magnesium is very low, cer-

tainly lower than that of cerium. This is in contradiction of the findings of Weibke and Schmidt<sup>13</sup> and of Haughton and Schofield<sup>12</sup> both of whom have reported essentially equivalent solid solubilities of cerium and of lanthanum in magnesium. The measurements on magnesium-lanthanum alloys were repeated after heat treating the alloys at 1100°F; no change was recorded in the conductivity after this heat treatment nor did any change occur upon



**Table 8 . . . Comparison of Properties of Magnesium-Rare Earth Alloys with Other Magnesium Alloys**

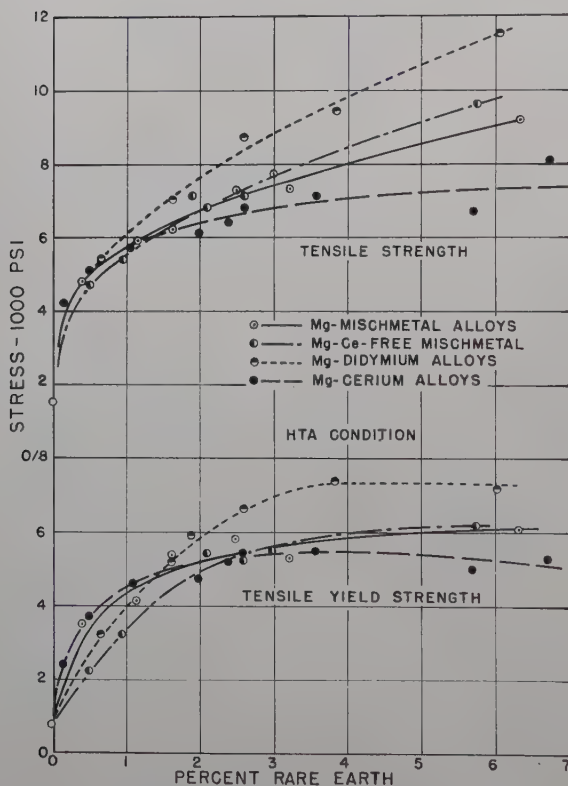
Alloy and Condition	Pct E	TYS*	TS*	Creep Limits* (100 hr)		
				0.1 pct Creep Extension	0.2 pct Total Extension	0.5 pct Total Extension
Room Temperature						
Mg + 6.33 pct Mischmetal (HTA).....	1.0	17.0	20.4			
Mg + 5.74 pct Ce-free Mischmetal (HTA).....	1.5	18.1	25.4			
Mg + 6.03 pct didymium (HTA).....	0.5	20.6	25.7			
Mg + 6Zn + 1Zr (HTA)†.....	4.9	23.9	39.5			
AZ92-HTA‡.....	2	23	40			
AZ63-HTS§.....	7	17	40			
400°F						
Mg + 6.33 pct Mischmetal (HTA).....	2.0	14.7	18.6	10.7	9.6	11.8
Mg + 5.74 pct Ce-free Mischmetal (HTA).....	4.5	16.8	24.3	13.5	10.3	15.5
Mg + 6.03 pct Didymium.....	3.2	19.2	27.7	14.7	11.2	17.0
Mg + 6Zn + 1Zr.....	21.5	15.1	18.1	4.1	4.3	6.0
AZ92-HTA.....	36.0	10.9	16.9	0.9		
AZ63-HTS.....		12.0	17.5	1.3	1.7	2.5

\* 1000 psi

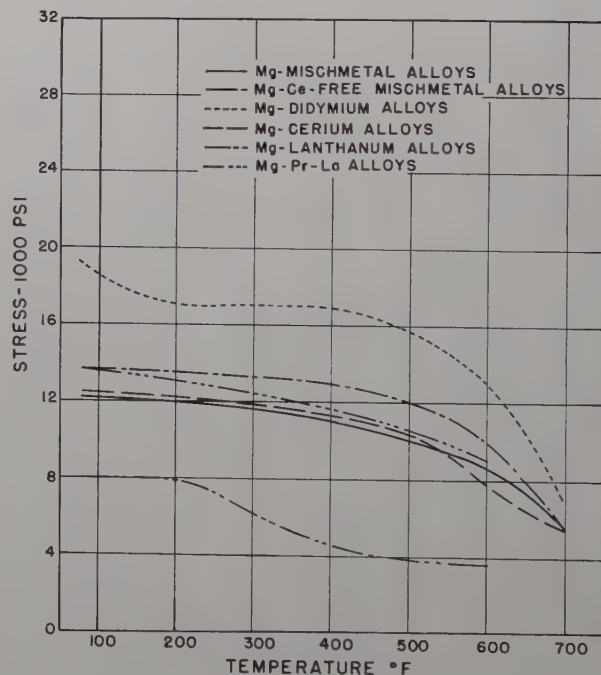
† Heat treated and aged at 400°F (16 hr)

‡ Mg-9Al-2Zn-0.2Mn:—Heat treated and aged at 425°F (10 hr)

§ Mg-6Al-3Zn-0.2Mn:—Heat treated and aged at 500°F (4 hr)



**FIG 13—Tensile yield strength and tensile strength at 700°F.**



**FIG 14—Tensile yield strength of magnesium—3 pct rare earth alloys as a function of temperature. HTA condition.**

aging at 400°F for 16 hr subsequent to the same heat treatment.

#### DENSITY

The density of magnesium-Mischmetal, magnesium-didymium, magnesium-cerium and magnesium-lanthanum alloys, all in the HTA condition, is shown as a function of composition in Fig 23. There is no significant difference in the density of these four types

of alloys. With this information on hand, it can be predicted that the density of magnesium-cerium-free Mischmetal alloys and that of magnesium-praseodymium-lanthanum will fall in the same range.

#### METALLOGRAPHY

Considerable attention was given to the microstructure of the alloys throughout this investigation; a few

micrographs (Fig 24 through 30) are presented to illustrate some of the most important features of the structures. The etchants used have been described by P. F. George.<sup>16</sup>

The as-cast structure of the 6 pct composition of each of the six alloy systems is shown in Fig 24 at a magnification of 100 × and in Fig 25 at 250 ×. The general distribution of second phase is clearly illustrated in Fig 24.

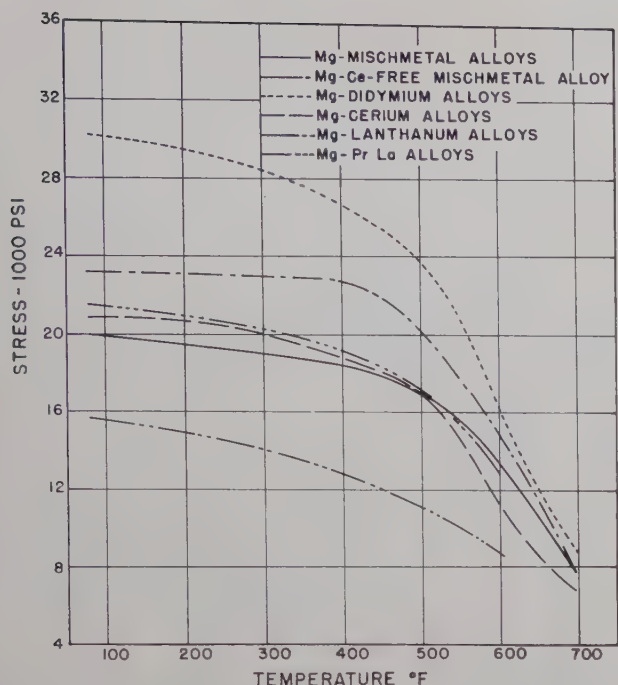


FIG 15—Tensile strength of magnesium—3 pct rare earth alloys as a function of temperature. HTA condition.

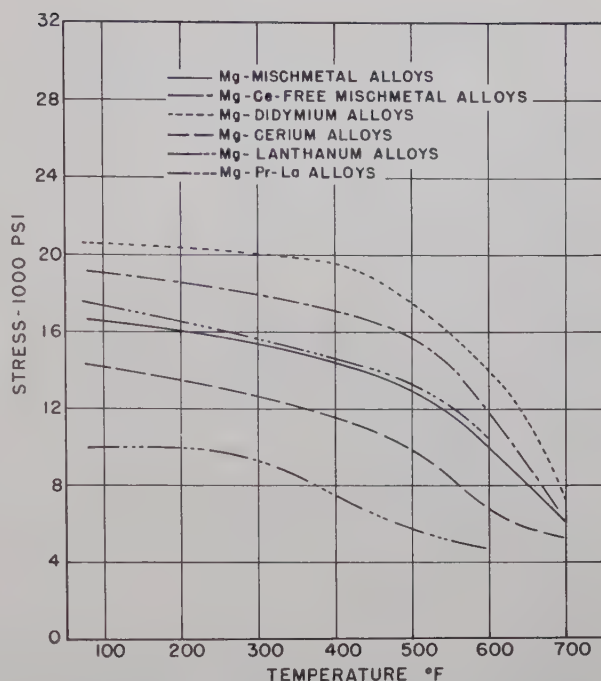


FIG 16—Tensile yield strength magnesium—6 pct rare earth alloys as a function of temperature. HTA condition.

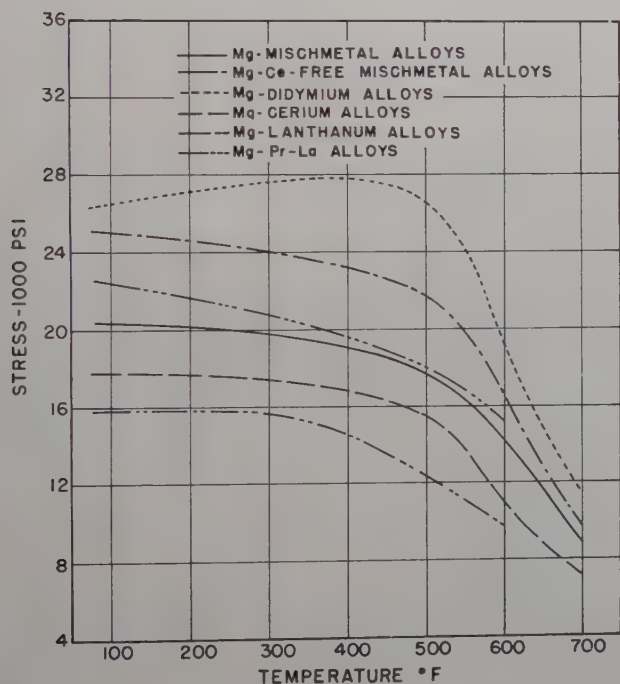


FIG 17—Tensile strength of magnesium—6 pct rare earth alloys as a function of temperature. HTA condition.

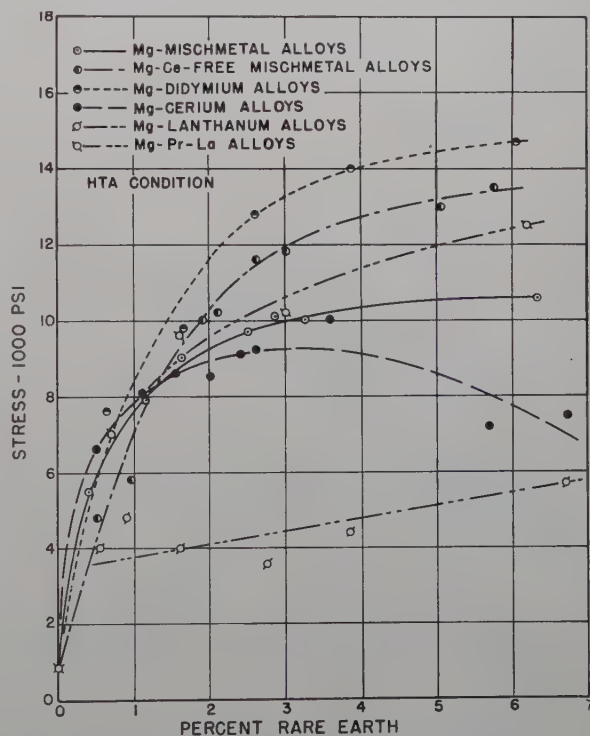


FIG 18—Limiting stress for 0.1 pct creep extension in 100 hr at 400°F.

All the alloys have a dendritic structure. There is a distinct difference in the nature of the eutectic in alloys containing more than one rare earth element from that in the magnesium-cerium alloy and in the magnesium-lanthanum alloy. Both the magnesium-cerium and the magnesium-lanthanum alloys have a very fine eutectic which can be resolved only with difficulty at

250  $\times$ . However, in all the other alloys, there is a very strong tendency for the eutectic to become divorced, in fact in magnesium-didymium alloys it is completely divorced, and what eutectic is present is in the form of massive compound areas with particles of magnesium solid solution imbedded in them. The magnesium-cerium and the magnesium-lanthanum eutectics are

shown more clearly at higher magnification in Fig 26 and 27 and for still better resolution of their structure the electron microscope was used with the results shown in Fig 28 and 29. That these two eutectics differ in the distribution of their respective components is apparent.

The structure of alloys containing lower amounts of rare earths are not



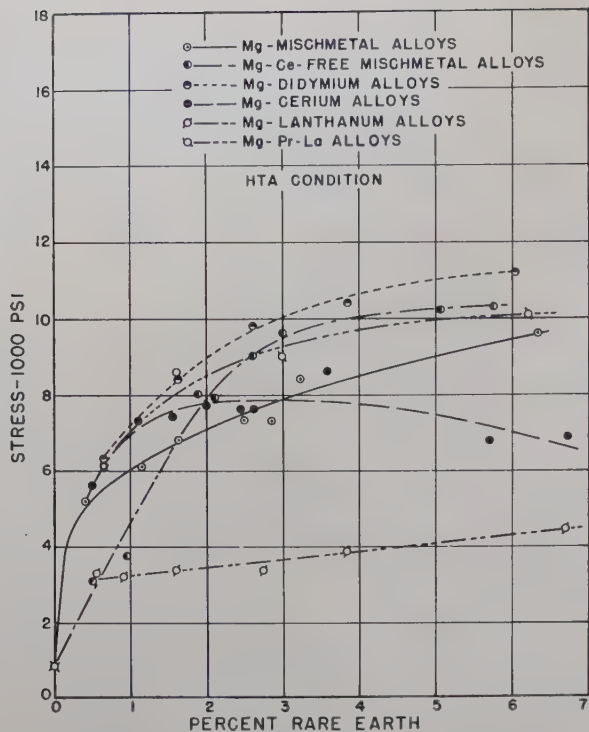


FIG 19—Limiting stress for 0.2 pct total extension in 100 hr at 400°F.

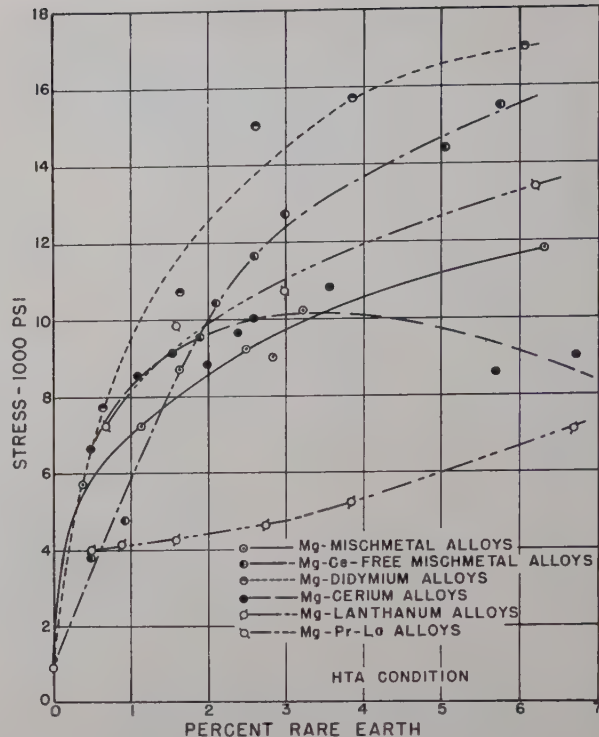


FIG 20—Limiting stress for 0.5 pct total extension in 100 hr at 400°F.

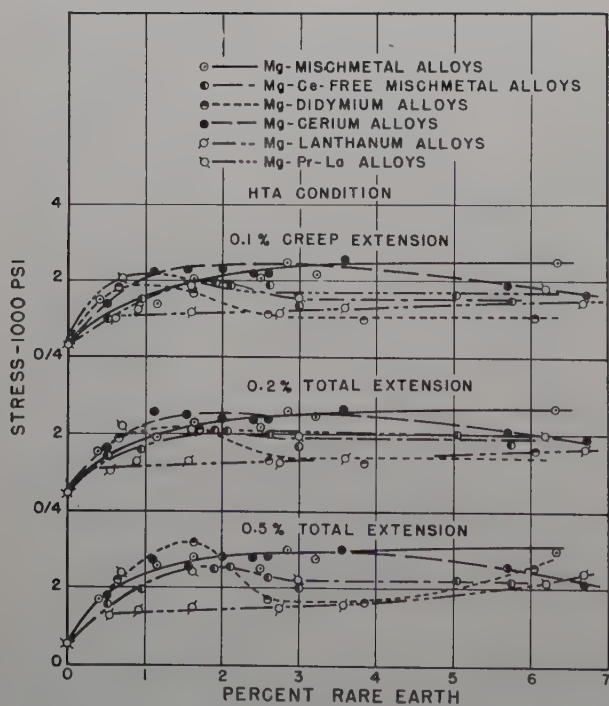


FIG 21—Limiting stresses for 100 hr creep at 600°F.

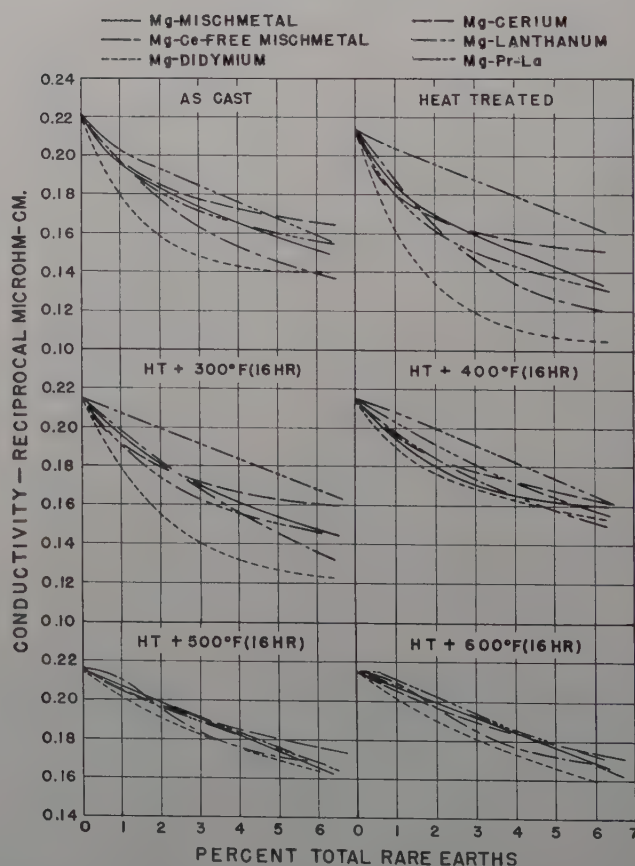


FIG 22—Electrical conductivity of magnesium—rare earth alloys at 95°F.

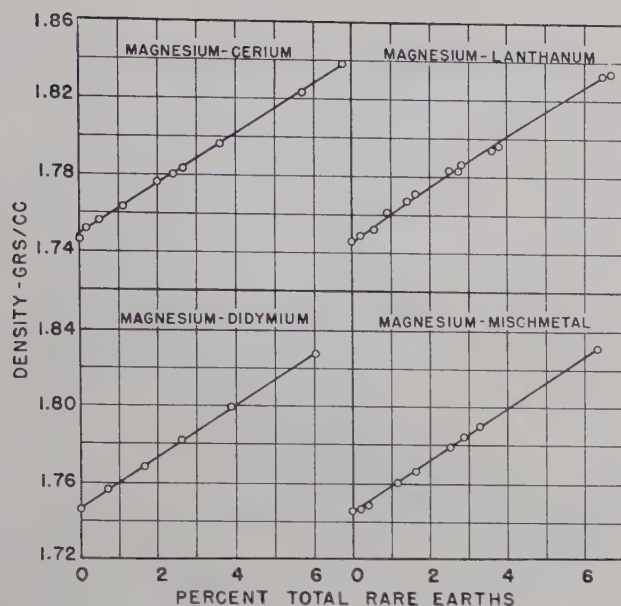


FIG 23—Density of magnesium—rare earth alloys at 25°C (77°F). HTA condition.

shown. The amount of eutectic decreases with decreasing alloy content and the eutectic network ceases to be the continuous phase in alloys containing less than 2 to 3 pct rare earth elements. The eutectic in the complex alloys is completely divorced and the magnesium-cerium and the magnesium-lanthanum eutectics show increasing tendency to divorce in lower alloy contents. There is compound or eutectic present in alloys containing as little as 0.10 pct total rare earths.

The microstructures of the 6 pct alloys after heat treating and aging are shown in Fig 30. In all the alloys the compound of the eutectic has been

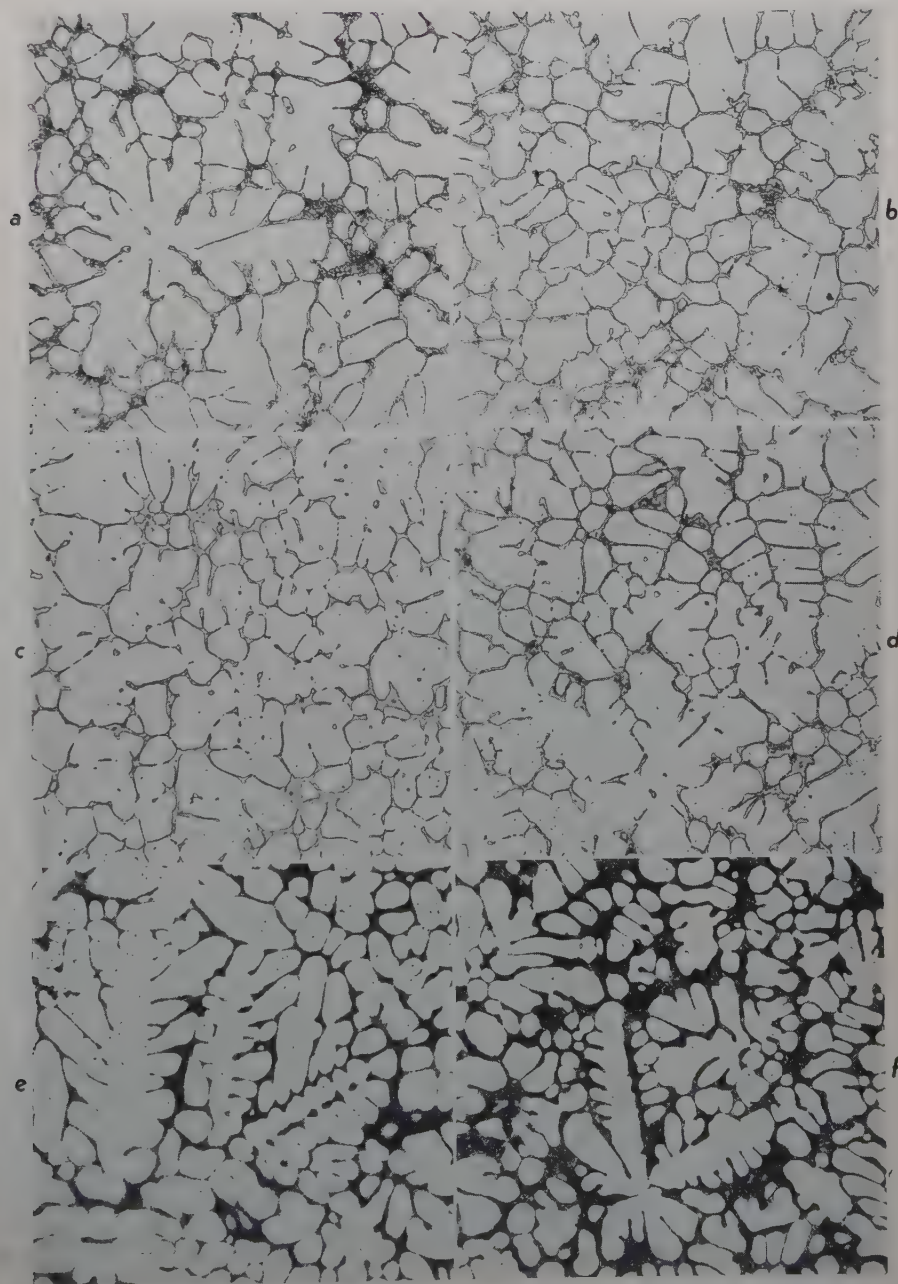


FIG 24—Structure of as-cast magnesium—rare earth alloys.

Glycol etchant. 100 X. Slightly reduced in reproduction. a. Mg + 6.33 pct Mischmetal. b. Mg + 5.74 pct Ce-free Mischmetal. c. Mg + 6.03 pct didymium. d. Mg + 6.19 pct (Pr + La). e. Mg + 6.73 pct cerium. f. Mg + 6.70 pct lanthanum.



coalesced and the continuity of the network has been broken so that the matrix is the continuous phase. For any given alloy content, the magnesium-didymium alloy has the least compound, and the magnesium-lanthanum alloy the most. The compositions of each alloy system which consists only of magnesium solid solution with no rare earth intermetallic compound after heat treatment are listed below:

Magnesium-Mischmetal.	0.18 pct total rare earth
Magnesium-cerium-free	
Mischmetal. ....	0.20 pct total rare earth
Magnesium-didymium..	0.65 pct total rare earth
Magnesium-praseodymium-lanthanum	None, lowest composition available was 0.69 pct
Magnesium-cerium. ....	0.15 pct total rare earth
Magnesium-lanthanum..	None, lowest composition examined was 0.16 pct

Increasing the heat treating time to 96 hr does not change these results.

Increasing the heat treating temperature for magnesium-lanthanum alloys does not decrease the amount of  $Mg_2La$  nor does it result in a completely homogeneous structure even in the lowest composition available. These findings confirm the conclusions formed from conductivity; neodymium has the greatest solid solubility in magnesium and lanthanum, the least.

Aging at 400°F for 16 hr produces a visible precipitate in the magnesium-cerium-free Mischmetal alloy but even here the amount of precipitate is small and the particles are very fine (Fig 30b). In a previous publication,<sup>4</sup> it has been shown that in the magnesium + 6 pct Mischmetal alloy in the HTA condition precipitate can be revealed only by the electron microscope.

Aging at 600°F produces a large amount of coarse precipitate in all the magnesium-rare earth alloys except magnesium-lanthanum, as shown previously for magnesium-Mischmetal alloys.<sup>4</sup> The time required to produce this precipitate is considerably longer in alloys containing 3 and 6 pct didymium than in all other alloys. Extending the aging time at 600°F to 5000 hr fails to produce any precipitate in magnesium-lanthanum alloys, whereas this treatment leads to the coalescence of the precipitate into fairly large particles in the case of magnesium-didymium and to the coalescence of the precipitate with the undissolved compound already present in magnesium-Mischmetal and magnesium-cerium alloys. Only magnesium-

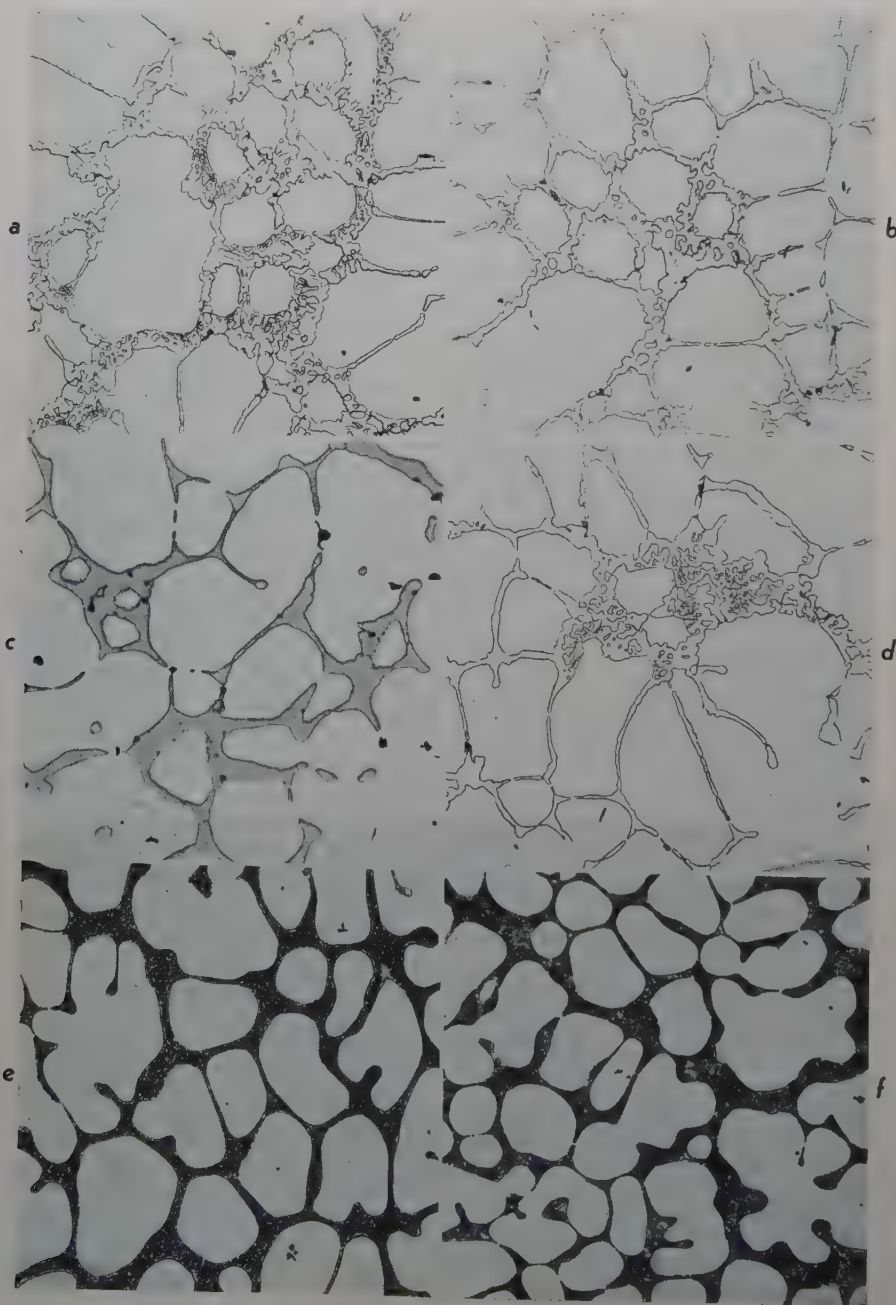


FIG 25—Structure of as-cast magnesium—rare earth alloys.

Glycol etchant. 250  $\times$ . Slightly reduced in reproduction. a. Mg + 6.33 pct Mischmetal. b. Mg + 5.74 pct Ce-free Mischmetal. c. Mg + 6.03 pct didymium. d. Mg + 6.19 pct (Pr + La). e. Mg + 6.73 pct cerium. f. Mg + 6.70 pct lanthanum.

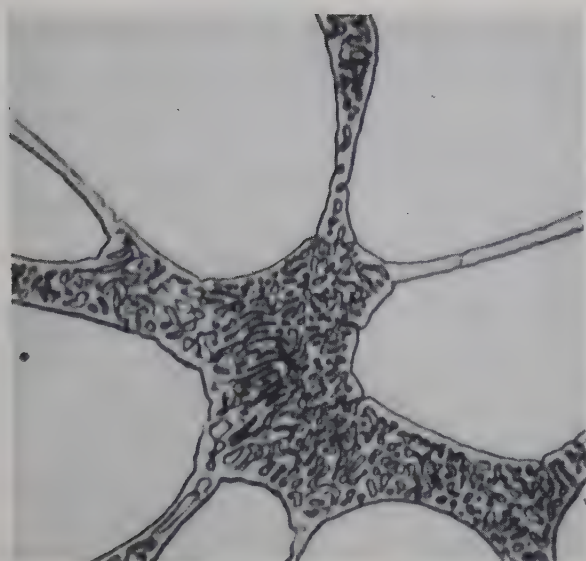


FIG 26 (left)—Eutectic in Mg + 6.73 pct Ce alloy. As-cast condition.  
Glycol etchant. 1000  $\times$ .

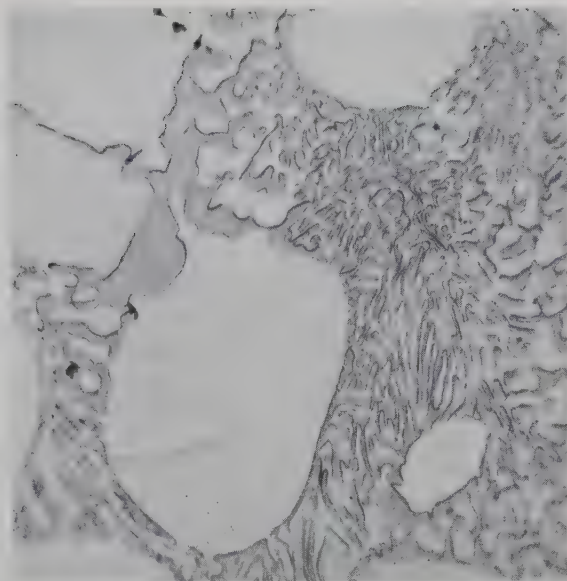


FIG 27 (right)—Eutectic in Mg + 6.70 pct La alloy. As-cast condition.  
Glycol etchant. 1000  $\times$ .

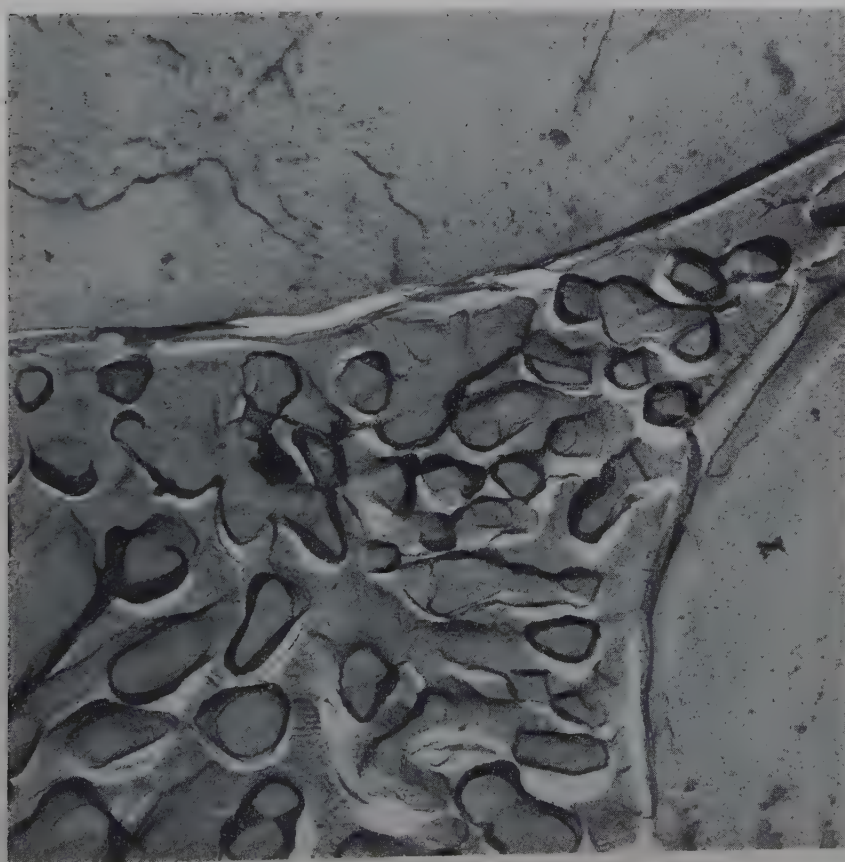


FIG 28—Electron micrograph of eutectic in Mg + 6.73 pct Ce alloy. As-cast condition.  
Methyl iodide etchant. 10,000  $\times$ .



cerium-free Mischmetal alloys retain a uniform distribution of precipitate after this prolonged aging treatment. This may explain the somewhat higher strength of these alloys after this treatment.

## Summary

All the rare earth metals investigated enhance the strength, hardness, and creep resistance of magnesium at room and elevated temperatures. There are, however, marked differences among the metals in the degree to which they improve these properties.

The various magnesium-rare earth alloys may be rated in the following order of decreasing tensile properties at room and elevated temperatures and creep resistance at 400 and 500°F:

1. Magnesium-didymium. 2. Magnesium-cerium-free Mischmetal. 3. Magnesium-praseodymium-lanthanum. 4. Magnesium-Mischmetal. 5. Magnesium-cerium. 6. Magnesium-lanthanum.

The relative effect of each rare earth metal on the creep resistance of magnesium at 600°F depends upon the composition level and, to a certain extent, upon the grain size.

Of the rare earth metals investigated, neodymium exhibits the greatest solid solubility in magnesium and lanthanum, the least. The solid solubility of lanthanum is much less than that reported in the literature. Cerium also appears to have a somewhat lower solid solubility in magnesium than that reported in the literature.

The results of this investigation have shown that considerably higher elevated temperature properties can be developed in magnesium-didymium and magnesium-cerium-free Mischmetal alloys than those exhibited by magnesium-Mischmetal alloys. At 400°F, for example, the properties of magnesium-didymium alloys are 20 to 50 pct higher than those of magnesium-Mischmetal alloys. Although the superiority of these alloys may not hold over the

entire temperature range, their advantage at certain temperatures may warrant their serious consideration for commercial applications. Further development of these alloys will depend upon the availability of the rare earths and a possible reduction in their cost.

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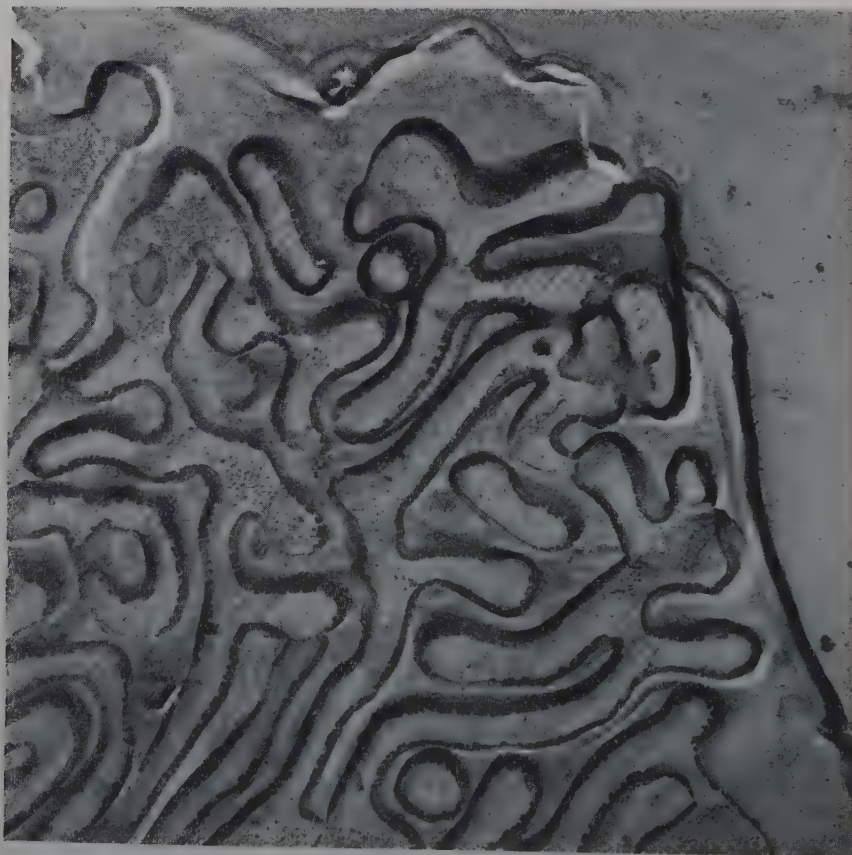


FIG 29—Electron micrograph of eutectic in Mg + 6.70 pct La alloy. As-cast condition. Methyl iodide etchant. 10,000 X.



**FIG 30—Structure of magnesium—rare earth alloys in the heat treated + aged condition.**  
Glycol etchant. 100 X. Slightly reduced in reproduction. *a.* Mg + 6.33 pct Mischmetal. *b.* Mg + 5.74 pct Ce-free Mischmetal. *c.* Mg + 6.03 pct didymium. *d.* Mg + 6.19 pct (Pr + La). *e.* Mg + 6.73 pct cerium. *f.* Mg + 6.70 pct lanthanum.

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|--|---|---|
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|  | 16. P. F. George: <i>ASTM Bull.</i> No. 129                       |   |



# The Relationship Between Electrical Conductivity and Composition of Molten Lead Silicate Slags

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Molten silicate salts, the important industrial byproducts termed "slags," are known to be electrolytic conductors at furnace temperatures. This property is due to their partial dissociation into ions with the ion kind and size being closely related to the slag composition. Ionic conductance, in liquids, is a function of the viscosity, which in turn is a function of the temperature. It could be anticipated, therefore, that a semi-log plot of conductivity vs. reciprocal absolute temperature might yield a straight line, in the case of molten silicates and salts. As liquids, the viscosity of these slags should follow the Shepard<sup>1</sup> relationship:

$$\log \text{viscosity} = \frac{A}{T} + B$$

Then, if the resistance of the molten slag is directly proportional to the viscosity:

$$\log R_s = \frac{A}{T} + B$$

Resistance measurements of molten halide salts,<sup>2</sup> and silicate salts<sup>3,4</sup> by various investigators have, indeed, shown this straight line relationship between resistance and temperature.

A further relationship between the

conductivity versus the degree of ionization, the size of the ions, and the slag composition is, also, to be expected. Large ions, and fewer ions, would move more slowly in a potential field and could not carry as much current as smaller, more numerous, and mobile, ions. Conductivity, therefore, would be smaller in the presence of such large ions and higher slag viscosities.

The conductivity-composition relationship is of considerable interest in the case of the industrial slags produced in iron blast furnaces, and non-ferrous smelting furnaces. These slags usually approximate three component systems with several minor components as impurities. At least one of the major

components is commonly purchased for the sole purpose of rendering the slag less viscous at a reasonable furnace temperature. Should the conductivity be related rather directly to the concentration of such a purchased slag component (that is, CaO) instrumental measurement and control at the furnace might be possible. Modern telemetric control methods in aqueous chemical process industries are often based on the property of conductivity of solutions, and its variation with composition.

Investigators<sup>3</sup> of the phenomenon of electrical conductivity in molten blast furnace slags have established that these systems show lines of equal conductivity on their ternary diagrams at one temperature level. These isoconductivity lines form closed contours on the diagrams, and at times, minimum areas occur at which these contours are essentially closed down to a point. The conductivity reading at such a point, at one temperature, would, of course, locate one on the diagram, and give the slag composition.

Research in this field is understandably scanty. The reactive nature of

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<sup>1</sup> References are at the end of the paper.

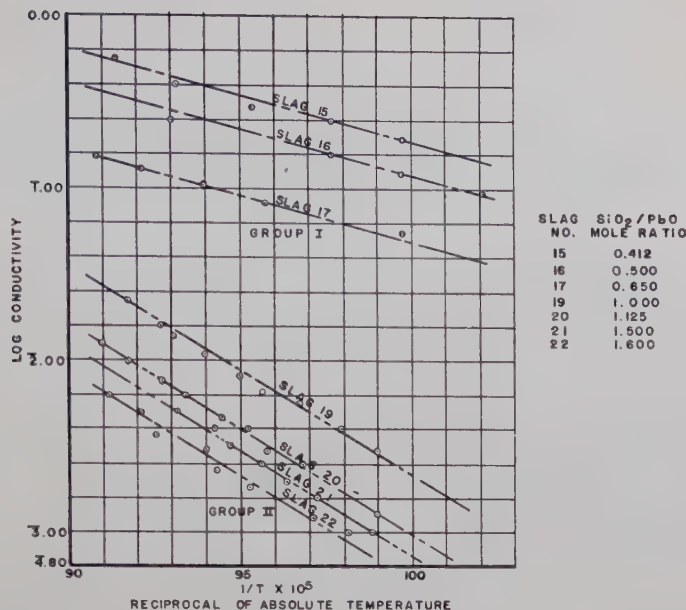


FIG 1—Log conductivity vs. reciprocal of absolute temperature for PbO-SiO<sub>2</sub> slags in liquid state.

these slag systems at their liquid temperatures renders the construction of a measuring apparatus extremely difficult. Temperature control is, also, not easy to achieve. Complex ternary systems often obscure experimental data by the introduction of uncontrollable variables.

## Experimental Procedure and Apparatus

The present research was started with the hope of obtaining some further data on industrial systems. However, preliminary work on low-melting lead silicate systems seemed like a logical starting point in establishing the validity of the conductivity-composition control method. Accordingly, several artificial slags were made up from cp litharge and silica. These ranged in content from 10 pct SiO<sub>2</sub> to 30 pct SiO<sub>2</sub>, by weight. These slags were melted in 10-g silica assay crucibles held in an electrically-heated pot furnace. An assembly, consisting of two platinum electrodes, plus a chromel-alumel thermocouple, was fitted on the top of the crucible. This allowed the electrodes and thermocouple to dip into the molten slag to a predetermined depth.

Temperature and conductivity were read simultaneously at each point; first with the temperature increasing, and then with it decreasing. Temperature was read on a Leeds and Northrup

portable potentiometer. Conductivity readings required 1000-cycle current, supplied by an audio-oscillator, and a resistance bridge circuit, calibrated in ohms. The null-point method was then used for both resistance and temperature. The conductivity apparatus was calibrated to yield specific conductivities, according to the formula:

$$L_s = \frac{K}{R} \quad \text{where } K \text{ is the cell constant and } R \text{ is the resistance reading in ohms.}$$

$K$  was determined by a calibration using standard KCl solutions.

## Results

A number of temperature-conductivity points were read for each slag composition, above its melting point. Fig 1 shows these points plotted to a log conductivity-reciprocal absolute temperature scale. It will be seen that the points for slag composition can be averaged as a straight line of formula:

$$\text{Log } L_s = \frac{-A}{T} - B$$

Where  $A$  is a constant for each line—the slope of the line—and  $B$  is a second constant.

This formula can be derived from the Shepard<sup>1</sup> formula for viscosity:

$$\text{Log } \mu = \frac{A}{T} + B$$

by assuming molten slag specific resistance as directly proportional to viscosity, whence:

$$\text{Log } R_s = \frac{A}{T} + B$$

and as,  $R_s = \frac{1}{L_s}$  then,

$$\text{Log } \frac{1}{L_s} = \text{Log } 1 - \text{Log } L_s = \frac{A}{T} + B,$$

$$\text{or, } \text{Log } L_s = \frac{-A}{T} - B.$$

The correspondence between this formula and the one for the plotted points in the lead silicate system is considered good evidence of the direct relationship between viscosity and conductivity in these slags.

It will be noted, also, that the four slags with SiO<sub>2</sub>/PbO ratios of 1.00, or greater, all have almost equal slopes. That is, for these compositions,  $A$  is a constant. Further, the three slags of SiO<sub>2</sub>/PbO ratios less than 1.00 yield lines with approximately equal slopes, or Constant  $A$ .

$A_2$  has an average value of 11,200, which makes the equation of the lines in group II:

$$\text{Log } L_s = \frac{-11,200}{T} - B$$

Similarly, the equation for group I is:

$$\text{Log } L_s = \frac{-5000}{T} - B$$

If we take unit conductivity ( $L_s = 1$ ) as a reference point, then for group II:

$$B = \frac{-11,200}{T_u} \quad \text{and} \quad T_u = \frac{-11,200}{B}$$

and for the group I:

$$T_u = \frac{-5000}{B}$$

where  $T_u$  is the absolute temperature of unit conductivity for each slag composition, corresponding to its constant  $B$ .



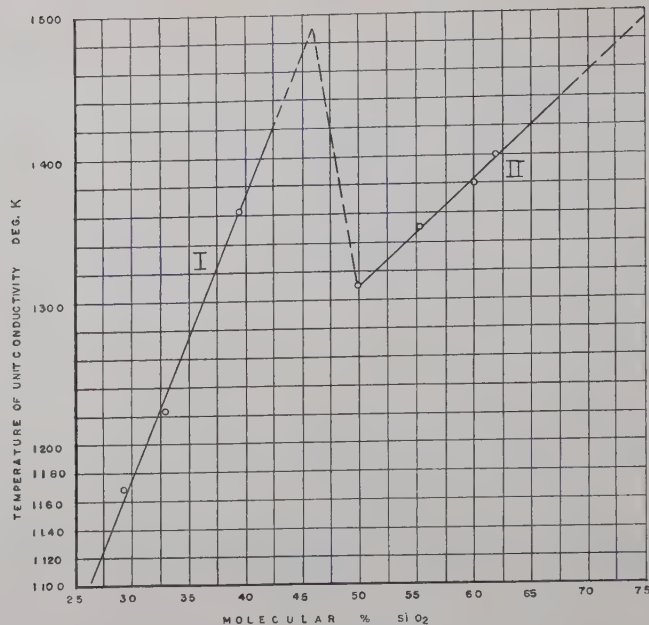


FIG 2—Temperature of unit conductivity vs. mol pct SiO<sub>2</sub>.

When these  $Tu$  values are plotted against molecular percentage of SiO<sub>2</sub>, the points can be averaged by two straight lines of different slopes, one for each group. This will be seen by a glance at Fig 2. The break between these two curves is seen to occur at about 50 mol pct SiO<sub>2</sub>, or about 21 pct by weight of SiO<sub>2</sub>. This break is such that the temperature of unit conductivity drops sharply at this composition. The slag suddenly conducts the same amount of electricity at a much lower temperature. This seems to indicate an increase in the number of ions and a decrease in the size of the ions, or both. Such an event could occur by the ionization, and disappearance, of a large molecular association such as PbO.SiO<sub>2</sub>. It is interesting to note that the constitutional diagram<sup>5</sup> of PbSiO<sub>3</sub> vs. temperature shows a temperature maximum in its liquidus line at 21 pct, by weight, SiO<sub>2</sub>. This maximum is at 1137 degrees absolute and corresponds to the compound PbO.SiO<sub>2</sub>.

The equation for line I, in Fig 2, is:  $Tu = 20N + 570$ , or

$$N = \frac{Tu - 570}{20} = 0.05Tu - 28.5$$

and as,  $Tu = \frac{-5000}{B}$  for group I

(when  $Ls = 1$ ) then  $N = \frac{-250}{B} - 28.5$   
 $=$  mol pct SiO<sub>2</sub> where  $B$ , for group I, is given by:

$$-B = \log Ls + \frac{5000}{T}$$

Similarly, for line II, in Fig 2:

$$N = \frac{Tu - 917}{7.8} = 0.128Tu - 117.6$$

and, substituting,  $Tu = \frac{-11,200}{B}$  for

group II, whence  $N = \frac{-1432}{B} - 117.6$

and,  $B$  for group II is given by:

$$-B = \log Ls + \frac{11,200}{T}$$

These equations can be used to predict the mol pct SiO<sub>2</sub> of lead silicate slags, in the absence of Fig 1 and 2, as follows:

Suppose that a slag of unknown silica content gave a specific conductivity reading of  $Ls = 0.00725$  mho at a temperature of 800°C or 1073°K. Here  $\log Ls = 3.860$ . Assume that any reading with a log value less than 2.00 will be in group II. Then,

$$-B = \log Ls + \frac{11,200}{T} = 3.860 + 10.42 = 8.28$$

$$\text{and } N = \frac{-1432}{B} - 117.6 = 173.2 -$$

117.6 = 55.6 mol pct SiO<sub>2</sub>. This is seen to be one of the points of slag 20, in group II. This slag therefore, has a composition of 25 pct SiO<sub>2</sub>, by weight.

For this simple binary system, these equations, or corresponding nomographs will convert specific conductivity, plus temperature readings to composition. Small amounts of impurities probably will not affect this relationship; however, this fact has not been definitely established. Extension of this method to other binary, and, perhaps, ternary systems would place a useful tool in the hands of the furnace metallurgist.

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# Cemented Titanium Carbide

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## Introduction

The increasing need for materials capable of withstanding higher operating temperatures for various applications such as gas turbine blading and other parts, rocket nozzles, and many industrial applications, has brought consideration of cemented carbide compositions. The well known usefulness of cemented carbides as tool materials is attributable to their ability to retain their strength and hardness at much higher temperatures than even complex alloys. However, it has been found that the temperatures encountered in cutting operations do not approach by several hundred degrees<sup>1</sup> those involved in the applications mentioned above where the interest is in materials possessing strength and resistance to oxidation at temperatures of 1800°F and above. At these latter temperatures, the tool type compositions which are made up essentially of tungsten carbide are found to oxidize very rapidly and to produce oxidation products of a character which offer no protection to the remaining body. As a further consideration, the density of the tungsten carbide type compositions is high, from about 8.0 to 15.0.

The refractory metal carbides as a class are the highest melting materials known as shown by Table 1 which summarizes the available data from the literature for the carbides of the elements which are sufficiently available for consideration for these uses. The density is also included in the table, since as mentioned above, it is an important consideration in many of the applications for which the materials would be considered. It has been established that in the tool compositions the mechanism of sintering with cobalt is such as to result in a continuous carbide skeleton and that the properties of the sintered composition are thus essen-

Table 1 . . . Melting Points and Density

Carbide	Melting Point °C	Density g per cc
TaC	3880	14.49
CbC	3500	7.82
TiC	3140	4.90
WC	2870	15.50
VC	2810	5.36
Mo <sub>2</sub> C	2380	8.9
Cr <sub>3</sub> C <sub>2</sub>	1890	6.68

tially those of the carbide.<sup>2</sup> On the hypothesis that this mechanism holds to a greater or less degree in cementing most of the refractory metal carbides with an auxiliary metal, it appears from Table 1 that titanium carbide compositions would offer possibilities for a high temperature material.

Titanium carbide has extensive use for supplementing the properties of tungsten carbide in tool compositions. Although the literature contains several references to compositions containing only titanium carbide with an auxiliary metal,<sup>3,4,5,6</sup> it may be inferred from the meager data that such compositions were deficient in strength and were considered to have poor oxidation resistance.<sup>7</sup> Kieffer, for instance, reports the transverse rupture strength of a hot pressed TiC composition at 100,000 psi as compared to up to 350,000 psi for WC compositions.

The work described herein was undertaken to determine the properties of compositions consisting of titanium carbide and an auxiliary metal and to improve the oxidation resistance of

such compositions. It appeared possible that the inclusion of one or more other carbides with titanium carbide might improve the oxidation resistance and also that this might be more desirable than other means from the point of view of maintaining the highest possible softening point. Consideration of the available carbides in Table 1 suggests tantalum and columbium carbides because of their high melting points and general refractoriness. The work on improving oxidation resistance was concentrated on the addition of tantalum carbide or mixtures of tantalum and columbium carbide. The auxiliary metals used included cobalt, nickel and iron. It was also desired to learn the general physical properties of these compositions.

## Experimental Procedure

The compositions used in this study were made by the usual powder metallurgy procedure applicable to cemented tungsten carbide compositions. The powdered carbide or carbides and auxiliary metal were milled together out of contact with air. In some cases cemented tungsten carbide balls and in other instances steel balls were used to eliminate any effect of tungsten carbide contamination. A temporary binder, paraffin, was then included in the mix and slugs or ingots were pressed with care to obtain as uniform pressing as possible. The ingots were presintered and the various shapes of test specimens were formed by machining, making the proper allowance for shrinkage during sintering. Thereafter the shapes were sintered in vacuum at temperatures of from 2800 to 3500°F. Final grinding to size was carried out by diamond wheels under coolant.

The titanium carbide used contained a minimum of 19.50 pct total carbon and a total of 0.50 pct metallic impurities as indicated by chemical and spectrographic analysis. It was found by X ray diffraction examination with

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TP 2725 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before April 1, 1950. Discussion is tentatively scheduled for publication in November 1950.

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† References are at the end of the paper.



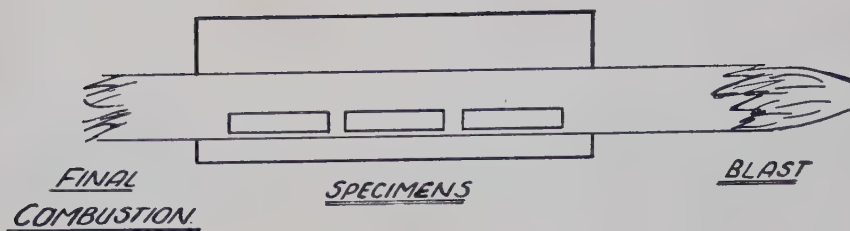


FIG 1—Arrangement of apparatus used for gas oxidation tests.

a Norelco Recording Spectrometer to have the usual NaCl type of cubic lattice with a parameter of 4.32 Å. The auxiliary metals were in the form of pure hydrogen reduced powders. The carbides used to modify the TiC for increasing its oxidation resistance were made by the McKenna "menstruum" process.<sup>8</sup> These were TaC and a three-carbide solid solution of the following approximate composition: Cb, 50.0 pct, Ta, 32.5 pct, Ti, 7.0 pct and carbon 10.5 pct. The latter is of interest because it offers a more available source of columbium and tantalum being made directly from suitable columbite ore without the need for separations.

Two tests were used to determine oxidation resistance. In the first test used in the earlier phases of the work a tube furnace arrangement combining electric and gas heating as shown in Fig 1 was used. The blast burner fuel gas ratio was so adjusted that combustion was not complete until the gases had passed over the specimens and through the tube and some flame was maintained at the exit end of the tube. An analysis of the gas at the specimens showed the following:

	Pct
CO.....	5.1
CO <sub>2</sub> .....	4.0
O <sub>2</sub> .....	6.1
N <sub>2</sub> and H <sub>2</sub> O.....	Balance

The temperature of the specimens was maintained at 2100°F. This condition was found to be insufficiently severe.

The second procedure consisted of heating the ground specimens on ceramic or carbon supports in an unsealed muffle furnace without atmosphere control. Various conditions with respect to time and temperature were used as noted later.

The problem of measuring deterioration is difficult. The scales or coatings formed are highly adherent and cannot be removed mechanically; and they cannot be removed chemically without the danger of attacking the sound body. Methods based on weight increase appear to be satisfactory although somewhat difficult of interpretation because of the varying stoichiometric increases associated with the oxidation

of the various constituents. Measurement of the increase in thickness was found to agree well with weight increase values and was used as a measure for most of the work. In a few instances, strength measurements made after exposure demonstrated the decrease in strength to be proportional to the thickness increase. In a few instances microscopic examination was also made on polished cross-sections.

The strengths were determined at room temperature and at 1800°F by the transverse bending method. The particular arrangement used is shown in Fig 2 and has been widely used for testing the strength of the tool compositions. It is satisfactory as long as the materials being tested are of high compressive strength so that the failure is by tension rather than by shear. It was found by a compressive-strength test using the method of Bridgeman<sup>9</sup> that the compressive strength of the composition of 80 pct TiC and 20 pct Co was 550,000 psi at room temperature and it was therefore considered that the short span specimen would yield satisfactory values for comparison purposes. This has since been confirmed qualitatively by work done on longer span specimens  $\frac{1}{4}$  by  $\frac{1}{2}$  by 4 in.<sup>10</sup> For the high temperature tests the specimen was heated by induction with a coil surrounding the supports and the specimen and with the thermocouple placed directly in contact with the bottom surface of the specimen. Additional tests were carried out at other temperatures up to 2000°F in the case of two of the compositions. The room temperature tests were made on a calibrated hydraulic testing machine or on a Brinell testing machine. The high temperature tests were made on a hand operated hydraulic press calibrated with a proving ring.

The density of the sintered compositions was measured in all cases to determine completeness of sintering. This was carried out on ground specimens using the water immersion method and with a commercial wetting agent in the water. The theoretical density was calculated on the assumption that

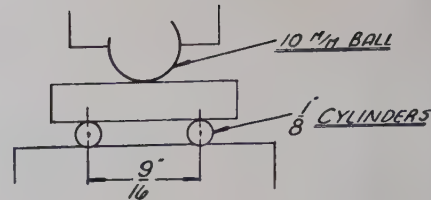


FIG 2—Arrangement for transverse rupture testing. Specimen cross section is 0.200 in. thick X 0.375 in. wide.

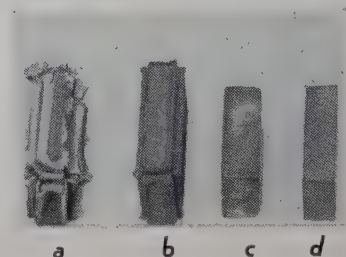


FIG 3—Comparison of oxidation of tungsten carbide and titanium carbide compositions.

a. 94 pct WC—6 pct cobalt. b. 73 pct WC—11 pct cobalt—16 pct titanium, columbium and tantalum as carbides. c. 80 pct TiC—20 pct cobalt. d. 60 pct TiC—20 pct cobalt—20 pct titanium, columbium and tantalum as carbides.

there was no retention of any of the carbides in solution in the auxiliary metal phase or change of any of the constituents in compositions. This was deemed a satisfactory criterion for determining completeness of sintering.

Other properties, including hardness, thermal and electrical conductivity and thermal expansion, were measured on two of the compositions to determine their general character.

## Experimental Results

Early oxidation tests with the apparatus shown in Fig 1 indicated the oxidation rate of TiC compositions to be as little as 0.0002 in. thickness increase per face per hr at temperatures up to 2100°F with an adherent coating formed. The transverse rupture strength at room temperature for the 80 pct TiC—20 pct Co composition was found to be from 160 to 190,000 psi and at 1800°F it was found to be of the order of 100,000 psi. Further oxidation tests made in the muffle furnace for 18 hr periods showed that while these TiC compositions oxidized somewhat rapidly at 1800°F (up to 0.0011 in. thickness increase per face per hr), they were considerably superior to the tungsten carbide base materials which showed rates as high as 0.050 in. and oxidation products of inferior character as shown by Fig 3. The first composition is 94 pct WC and 6 pct Co, the

second 73 pct WC and 11 pct Co and the balance titanium, tantalum and columbium as carbides. In both instances the oxides are fluffy and non-adherent. The third specimen is 80 pct TiC and 20 pct Co. The fourth composition is the same as the third except that 20 pct of the solid solution of columbium and tantalum and titanium carbides was included as will be discussed later.

The oxidation rates of compositions containing varying quantities of cobalt, nickel and iron with TiC only and of compositions including TaC and the three carbide solid solution were determined in an 18 hr muffle test as shown in Table 2. The rates for the various auxiliary metals are observed to be of the same order of magnitude. In these preliminary tests it was found that from 25 to 35 pct of TaC substituted for an equal weight of TiC reduced the oxidation by tenfold as is shown in Table 2. This same result could be accomplished equally well by smaller percentages of the columbium, tantalum, titanium carbide solid solution. A series of compositions was therefore prepared using cobalt as the auxiliary metal maintained at 12.3 pct by volume of the composition. The three carbide solid solution content was varied as shown in Fig 4. The sintered test pieces were subjected to a 64 hr test in the muffle with the results shown. The marked effect of the columbium and tantalum in even small percentages in reducing the oxidation is to be noted as well as the fact that compositions which are very high in columbium and tantalum carbide oxidize more rapidly than the high TiC

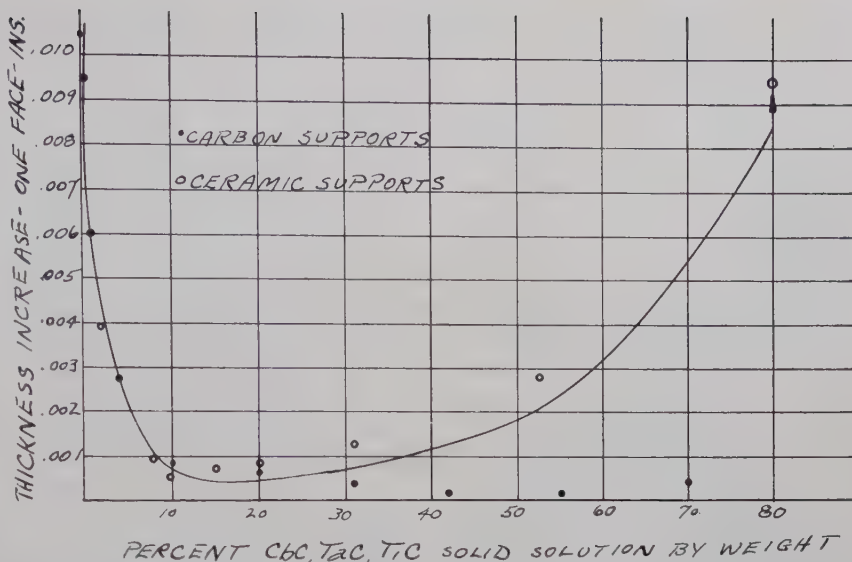


FIG 4—Effect of CbC, TaC, TiC solid solution on oxidation of TiC compositions. 64 hr at 1800°F in open muffle furnace.

compositions. Two tests were made in one of which the specimens were supported on ceramic boats and in the other on carbon supports. The difference in the results should be noted.

A 210 hr oxidation test at 1800°F in the open muffle on a few selected compositions showed substantially the same results as the 64 hr test as shown in Table 3. Fig 5 shows the character of coating formed on the composition containing 18.7 pct Co, 15 pct three carbide solid solution and 66.3 pct TiC after heating for 210 hr at 1800°F in the open muffle. The overall coating thickness is approximately 0.003 in. A short test of 18 hr at 2200°F showed relative oxidation rates to be unchanged with the composition contain-

ing 20 pct of the three carbide solid solution being highly resistant. The thickness increase was 0.0182 in. for the 80 TiC-20 Co composition and 0.0014 in. for the equivalent composition containing 15 pct solid solution.

A study was made to determine whether any change in the rate of oxidation occurred with time with the results shown in Fig 6. The two compositions used were the 80 pct TiC-20 pct Co composition and the similar composition containing 15 pct of the columbium, tantalum, titanium solid solution. It is observed that within the accuracy of the measurements both compositions show straight line though widely varying rates of oxidation.

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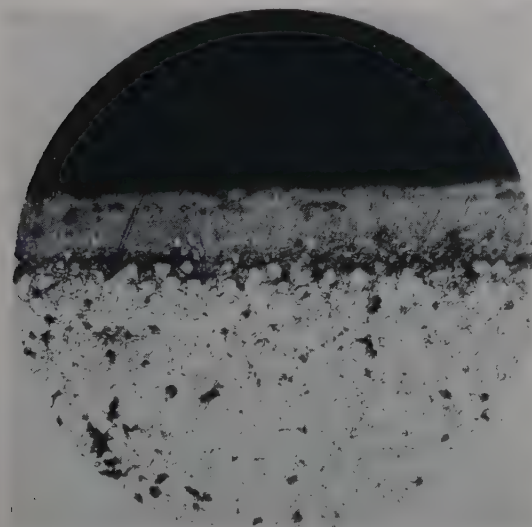


FIG 5—Coating on TiC composition heated 210 hr in muffle at 1800°F. 66.3 pct TiC, 15 pct solid solution of CbC, TaC, and TiC, 18.7 pct Co. 100 X.

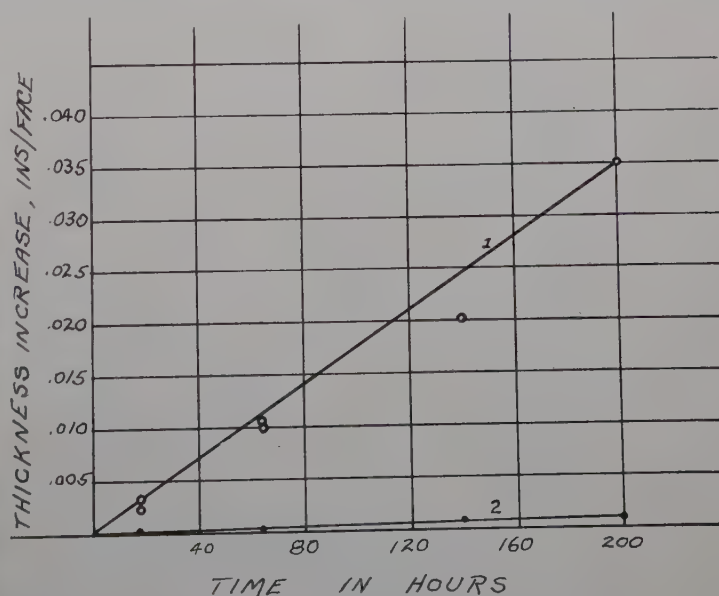


FIG 6—Variation of rate of oxidation of TiC composition with time. (1) 80 pct TiC—20 pct cobalt. (2) 66.3 pct TiC—15 pct solid solution of CbC, TaC and TiC.



**Table 2 . . . Oxidation of Various TiC Compositions**

**18 hr at 1800°F—Muffle Test**

Composition Ingredients and Pct	Increase in Thickness In. Per Face
95 TiC, 5 Co.....	0.0021
90 TiC, 10 Co.....	0.0025
80 TiC, 20 Co.....	0.0022
70 TiC, 30 Co.....	0.0038
90 TiC, 10 Ni.....	0.0015
85 TiC, 15 Ni.....	0.0031
80 TiC, 20 Ni.....	0.0028
80 TiC, 20 Fe.....	0.0040
60 TiC, 25 TaC, 15 Ni.....	0.0003
50 TiC, 35 TaC, 15 Ni.....	0.0001
71 TiC, 10 S.S.* 19 Co.....	0.0006
62 TiC, 20 S.S., 18 Co.....	0.0003

\* S.S. refers to the solid solution of columbium, tantalum and titanium carbides.

**Table 3 . . . Oxidation Test of TiC Compositions**

**210 hr at 1800°F—Muffle Test**

Composition Ingredients and Pct	Increase in Thickness In. Per Face
80 TiC, 20 Co.....	0.0359
66.3 TiC, 15 S.S.* 18.7 Co.....	0.0009
51.7 TiC, 31 S.S., 17.3 Co.....	0.0009
41.8 TiC, 41.8 S.S., 16.4 Co.....	0.0019
66.3 TiC, 15 S.S., 18.7 Ni.....	0.0020

\* S.S. refers to the solid solution of columbium, tantalum and titanium carbides.

carbide solid solution upon the room temperature strength was determined upon the same series of compositions as used to obtain the oxidation data of Fig 4. The results are shown in Fig 7. The consistent effect of the columbium and tantalum in reducing strength is to be noted. The strengths at room temperature and 1800°F of various other compositions are given in Table 4. It is to be noted that although the strengths of various TiC-Ni compositions are greater at room temperature than those of the TiC-Co compositions, the strength decreases more rapidly with temperature so that at 1800°F, they are somewhat weaker. The compositions using iron show promise. It has been suggested that, since TiC

**Table 4 . . . Strength and Density of Various TiC Compositions**

Composition No.	TiC Pct	Other Carbide Pct	Auxiliary Metal Pct	Transverse Rupture psi X 1000		Density	
				Room Temp.	1800°F	Theoretical	Actual
1	95		Co 5	126		5.12	5.26
2	90		Co 10	122		5.23	5.23
3	80		Co 20	160	111	5.47	5.42
4	65		Co 35	152		5.91	5.84
5	90		Ni 10	123		5.23	5.26
6	85		Ni 15	151	90	5.35	5.40
7	80		Ni 20	173	91	5.47	5.45
8	70		Ni 30	154		5.61	5.60
9	90		Fe 10	124		5.19	5.11
10	80		Fe 20	136		5.42	5.32
11	60	TaC 25	Ni 15	85	71	6.51	6.52
12	50	TaC 35	Ni 15	93	42	7.08	7.02
13	66.3	S.S.* 15	Ni 18.7	147	82	5.85	5.78

\* S.S. refers to the solid solution of columbium, tantalum and titanium carbides.

compositions were known to have inferior strengths, the strengths obtained in this work were the result of approximately 3 pct of tungsten resulting from the milling of these compositions with cemented tungsten carbide balls. Accordingly, a composition was prepared containing 80 pct TiC and 20 pct Co wherein steel balls were used for the milling. Analysis showed this composition to contain only spectrographic traces of tungsten. The room temperature transverse rupture strength of this composition averaged 153,000 psi as compared with 160,000 with the same composition milled with tungsten carbide balls.

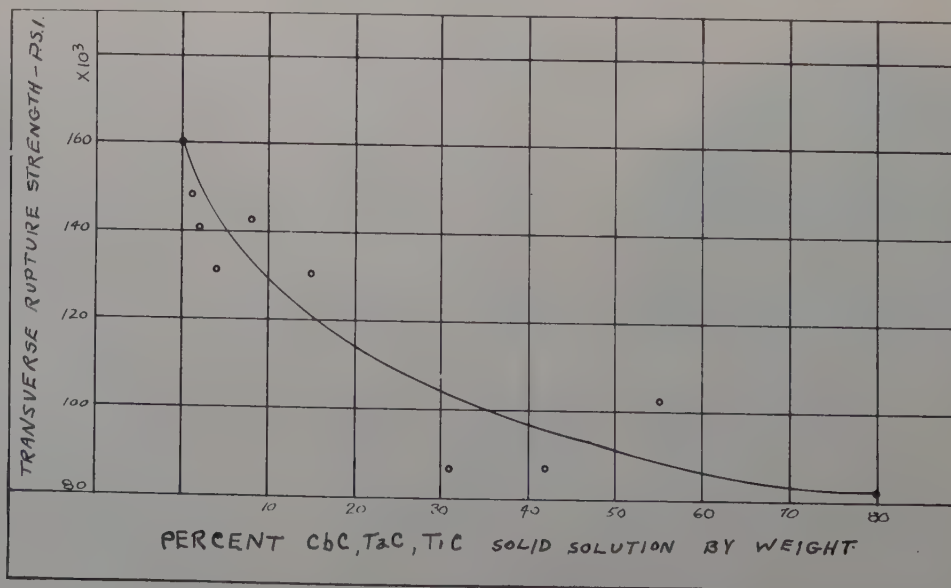
The elevated temperature strengths of two of the compositions, that containing 80 pct TiC and 20 pct Co and the equivalent containing 15 pct three carbide solid solution are shown in Fig 8 for temperatures up to 2000°F. The slow rate of falling off of strength is to be noted. The lower rate of decrease

of the composition containing the columbium and tantalum carbide is of note. Table 4 also includes some high temperature modulus of rupture values for some of the other compositions.

The theoretical and actual densities have been shown in Table 4 and it will be noted that the actual densities correspond very closely.

## Discussion of Results

The most notable result is the marked decrease in oxidation of TiC compositions effected by the minor addition of TaC or of CbC and TaC in the form of the three carbide solid solution. This reduction in the amount of oxidation ranged up to 40 times in some instances. The effect cannot be accounted for on the basis of the oxidation characteristics of the additive carbides since, of themselves, the other carbides do not possess the oxidation resistance of



**FIG 7—Effect of increasing percentages of CbC, TaC, TiC solid solution upon strength of TiC compositions, 12.3 pct Co by volume throughout.**

TiC. The explanation which would appear to hold is that the ratio of the oxides in the oxidation product and their character must be such as to form an adherent continuous film and thus prevent further access to the carbide structure. Although no tests were conducted on the effect of CbC alone as an additive to TiC compositions, the combined effect of the columbium and tantalum carbides contained in the three carbide solid solution indicates that the columbium contributes considerably to the protection obtained.

The use of various auxiliary metals has little effect on the oxidation rate and as a secondary observation the percentage of auxiliary metal has small influence on the oxidation rate. It thus appears that the rate of oxidation is largely determined by the carbide constituents.

The data on modulus of rupture show in general that titanium carbide compositions may have strengths as much as 75 pct higher than those previously reported. Although these strengths are considerably below the general level of those of the cemented tungsten carbide compositions, the cemented titanium carbide compositions are strong materials. A better basis upon which to compare these materials with other materials, at least with alloys, is tensile strength and work upon this phase is now being carried out. However, based on the work of a number of laboratories the short time tensile strength of brittle materials may be expected to be from 0.4 to 0.6 the modulus of rupture value.<sup>10</sup> This would make the high temperature tensile

strengths of cemented titanium carbide comparable with the best alloys and ceramics.

The marked difference in the rate of oxidation observed between the gas fired tube furnace and the muffle furnace, which was the equivalent of the differences found with and without columbium and tantalum carbides, emphasizes the effect of variations in the atmosphere upon oxidation results. The importance of even small variations in atmosphere upon oxidation results is shown by the results in the muffle furnace when ceramic vs. carbon supports are used for the specimens. Such differences in the effect of furnace atmosphere are insignificant in the case of compositions which have high oxidation resistance as shown by Fig 4.

## Other Properties

It is of interest to know other properties of these TiC compositions, some of which are summarized in Table 5 for two of the compositions.

Table 5 . . . Additional Properties of TiC Compositions

	Pct 80 TiC 20 Co	Pct 66.3 TiC 15.0 S.S.* 18.7 Co
Young's Modulus of Elasticity		
Room Temperature.....	$55.0 \times 10^6$ psi	$57.3 \times 10^6$ psi
Hardness, RA, Room Temperature.....	90.5	89.5
Thermal Expansion per °F 100 to 1800°F.....	$5.0 \times 10^{-6}$	$4.5 \times 10^{-6}$
Thermal Conductivity cal per sec per °C per cm.....	0.085	0.075
Electrical Conductivity Per Cent of Copper Standard.....	5.0	4.0

\* S.S. refers to the solid solution of columbium, tantalum and titanium carbides.

## YOUNG'S MODULUS OF ELASTICITY

These values were determined by the sonic vibration method<sup>11</sup> using ground bars  $\frac{1}{4} \times \frac{1}{2} \times 3$  in. Comparisons were made with cemented tungsten carbide bars. These high moduli are indicative of the formation of a continuous carbide skeleton as in the case of the tungsten carbide compositions.

## HARDNESS

These values were determined using a special Rockwell A Brale which was calibrated upon a cemented tungsten block calibrated by the Wilson Mechanical Instrument Co.

## THERMAL EXPANSION

These values were determined by the use of a Bureau of Standards type dilatometer. Virtually straight line expansion curves were obtained. While these values are somewhat higher than the general level of those for cemented tungsten carbide compositions, they

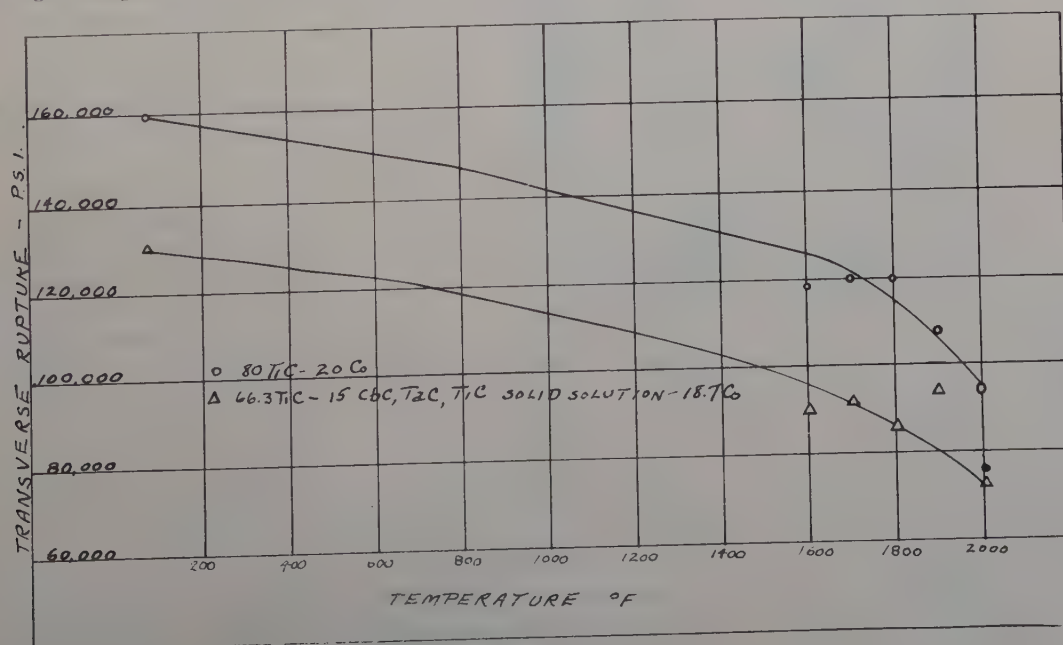


FIG 8—Effect of temperature upon the strength of TiC compositions.



iron and lead. The conductivity of the cemented titanium carbide compositions is somewhat below that of the tungsten carbide compositions although above the level of highly alloyed ferrous base materials and many times higher than ceramics. The thermal conductivity of a material has a large bearing on its ability to resist thermal shock resulting from rapid temperature changes.

## ELECTRICAL CONDUCTIVITY

These values were obtained by the Kelvin Bridge method using ground bars  $\frac{1}{4}$  in. diam by 12 in. long and calibrating against a 0.1 ohm standard accurate to  $\pm 0.02$  pct.

## Structure

Fig 9 and 10 show the etched structures of two of the compositions at 1500 magnifications which is largely used for examining cemented tungsten carbide tool compositions. The polishing is done with diamond dust using lead-tin laps as in the case of tungsten carbide compositions but greater care is needed to avoid scratches because of the lower abrasion resistance of these compositions. The 2 pct nital etch has been found to produce preferred etching to the conventional alkali ferri-cyanide etch used on tool compositions.

The structures are comparable to those of the tungsten carbide compositions in that they show two phases, the carbide phase and the phase which is predominately auxiliary metal. The grains are much more rounded than is the case with tungsten carbide compositions. The effect of CbC and TaC in causing larger grain size is to be noted. A specimen prepared for the composition of Fig 10 after 210 hr exposure at 1800°F is shown in Fig 11. There appears to be no significant change in structure.

X ray diffraction examination of the compositions of Fig 9 and 10 with the Norelco Recording Spectrometer shows that in both cases only a single carbide phase is present. In the case of the TiC-Co composition the TiC phase has a lattice parameter of  $4.32 \text{ \AA}$  as for the TiC used in making the composition. This indicates that no more than a small quantity of Co could be held in solution in the TiC. The lattice parameter of the composition containing titanium, columbium and tantalum carbides was found to be  $4.34 \text{ \AA}$  indicating a complete solid solution, as would be expected from the work of Norton and Mowry.<sup>13</sup> The amount of

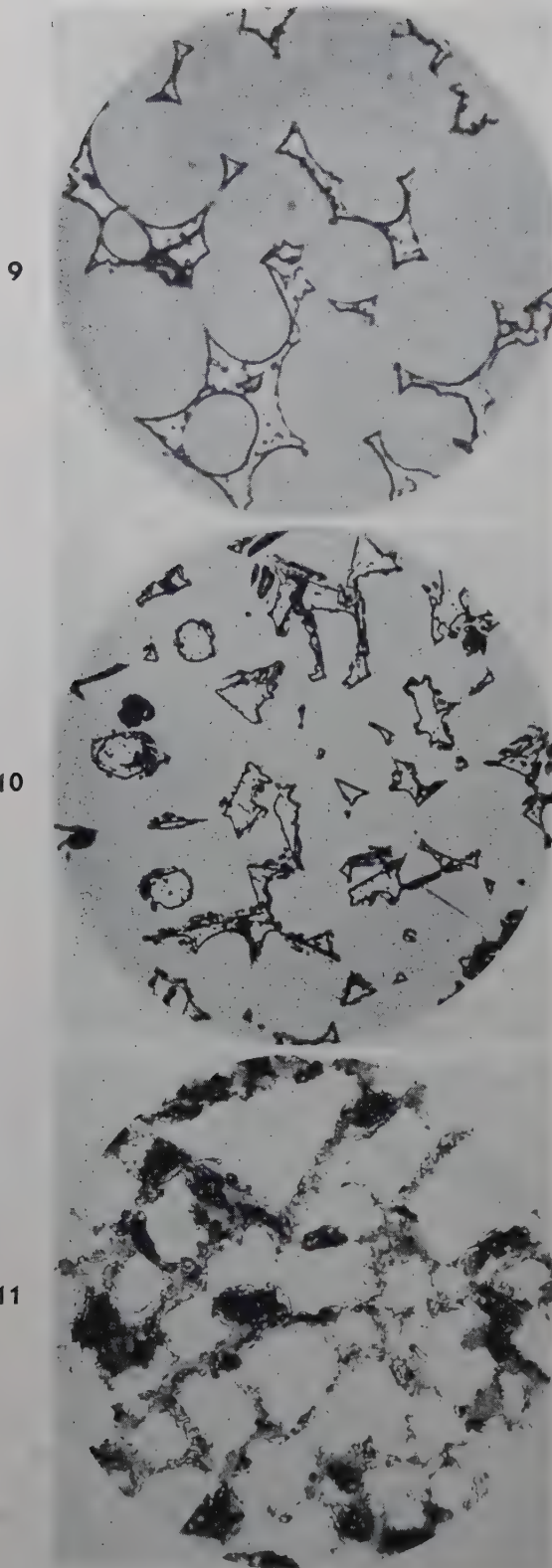


FIG 9—Structure of cemented TiC—80 pct TiC, 20 pct Co.  $\times 1500$ , nital etch.

FIG 10—Structure of cemented TiC—66.3 pct TiC, 15 pct CbC, TaC, TiC solid solution, 18.7 pct Co.  $\times 1500$ , nital etch.

FIG 11—Composition of Fig 10 after 210 hr at 1800°F.  $\times 1500$ , nital etch.

are one half or less those for ferrous base alloys and of the order of those for ceramics. The thermal expansion is an important consideration in the resistance of materials to thermal shock.

## THERMAL CONDUCTIVITY

These values were obtained by the method described by McKenna.<sup>12</sup> The apparatus was standardized on pure

TiC retained in the Co phase in the sintered compositions has not yet been determined.

## Summary

This work has shown that cemented titanium carbide compositions prepared by powder metallurgy methods have transverse rupture strengths up to 175,000 psi or 75 pct greater than previously reported in the literature. Either cobalt, nickel or iron may be used as an auxiliary metal. The two former appear very nearly equivalent in titanium carbide compositions. Iron is somewhat inferior.

Compositions containing only titanium carbide and an auxiliary metal are considerably superior to the tungsten carbide compositions with respect to the character of the oxidation products. It has also been found that the addition of minor percentages of tantalum carbide or of columbium and

tantalum carbide reduces the oxidation rate of the titanium carbide compositions to very small values at temperatures up to 2200°F.

The general properties of cemented titanium carbide compositions are comparable with those of the cemented tungsten carbide compositions. When the strength, oxidation resistance, thermal expansion and conductivity are considered, these compositions offer excellent possibilities as high temperature materials.

## Acknowledgment

The authors acknowledge the work of J. R. Bridge in obtaining the data upon the general properties of these materials and of others in this laboratory for their assistance in this work.

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## Binary Chart for Interconversions of Mol, Weight, and Volume Percent

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The accompanying Fig 1 is a graphical scheme of intermediate accuracy for expediting interconversions of mol, weight, and volume percents. This chart consists of a family of curves of mol or atomic or volume percent plotted against weight percent, each curve in the family providing for a certain ratio of molecular or atomic weights or densities. Thus, the whole range of variations is presented in a very familiar format, and the conversion is effected simply by choosing the proper curve and reading off the required values. An entire series of conversions for a given binary system can be found by following the appropriate curve.

It is interesting to examine the analytical geometry of the plots. The curves, which must fit into a square

and all join at the 0 and 100 pct points, may be shown to be hyperbolas with their centers on the extension of the diagonal of the square which they intersect. Multiplying out the expression

$$\text{atomic fraction of } A = y = \frac{\frac{x}{A}}{\frac{x}{A} + \frac{1-x}{B}}$$

where  $x$  is the weight fraction of  $A$ , and  $A$  and  $B$  are atomic weights, we get

$$xy + \frac{A}{B}y - \frac{A}{B}xy = x$$

Upon rotating and translating axes and

avoiding imaginaries, it is found that the angle of rotation is  $-45^\circ$ , the origin

$$\text{moves up } \frac{\sqrt{2}}{2} \text{ units and } \frac{\sqrt{2}}{2} \left( \frac{\frac{A}{B} + 1}{\frac{A}{B} - 1} \right)$$

units to the right, and the standard form is

$$\frac{x^2 - y^2}{\frac{1}{2} \left[ \left( \frac{\frac{A}{B} + 1}{\frac{A}{B} - 1} \right)^2 - 1 \right]} = 1$$

Thus the curves are equilateral hyperbolas, their centers are

$$\sqrt{\frac{1}{2} \left[ \left( \frac{\frac{A}{B} + 1}{\frac{A}{B} - 1} \right)^2 - 1 \right]}$$

units away from their intersection with

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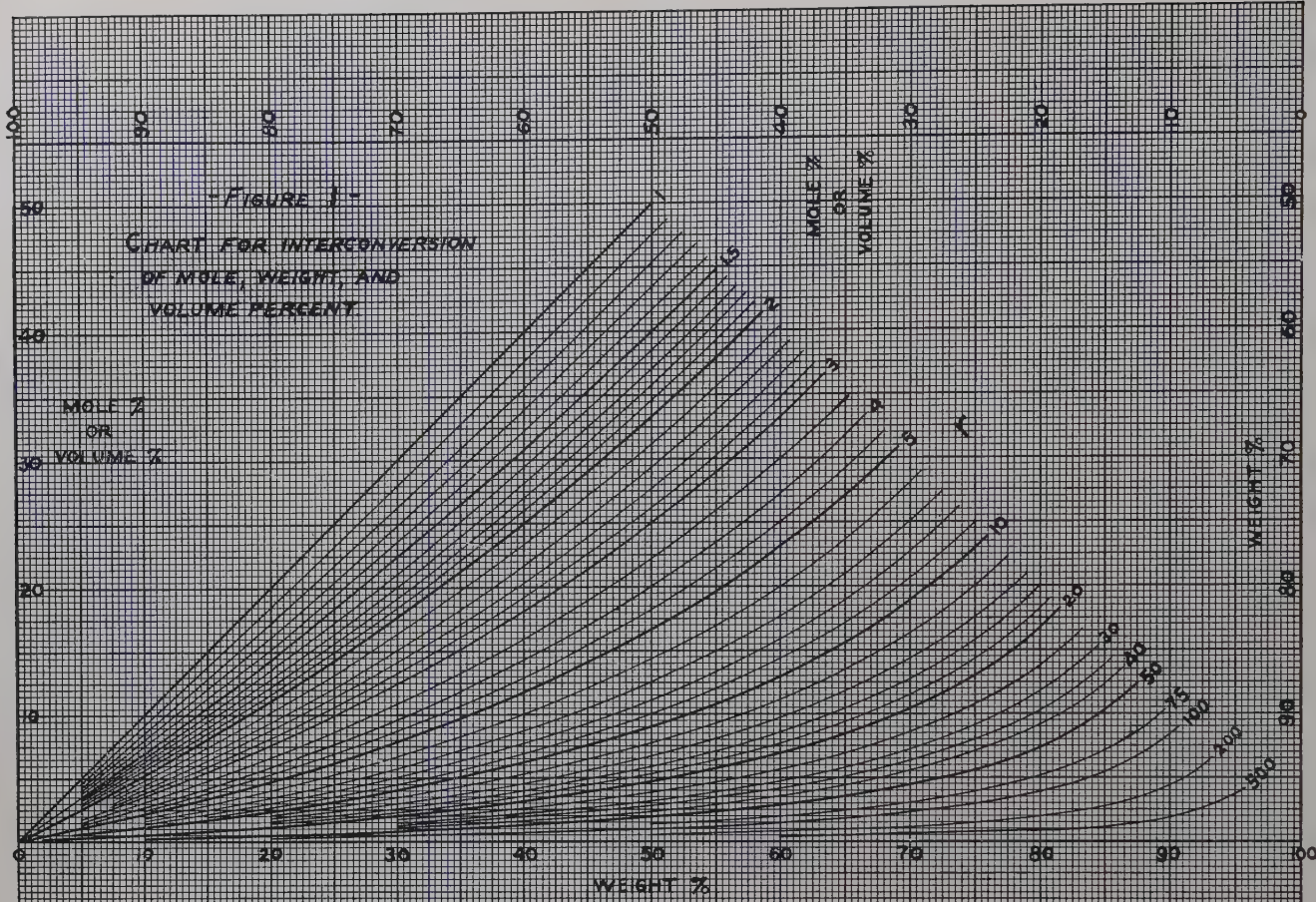


FIG 1—Chart for interconversion of mol, weight, and volume percent.

the diagonal and are on that diagonal, and the curves are symmetrical with respect to that diagonal. As the ratio of  $A:B$  increases, both the center of the curve and the intersection of the curve with the diagonal approach the lower right hand corner of the square plot from opposite sides.

In the actual chart only one triangular quarter of the entire square is shown. Elimination of half of the square is accomplished by setting up the rule that the larger ratio always be taken, in other words, by making  $A$  the component with the larger atomic or molecular weight. Then, since the curves are symmetrical, only half of the remaining plot need be drawn, and the diagram may be turned as required. The scales are labeled so as to prevent error.

A few examples of the use of the chart may be useful. As may be seen on the chart, the atomic or molecular weight ratio, or density ratio is called  $r$ . Suppose it is desired to convert 20 wt

pct of Ag in the Ag-Cu system to atomic percent Ag. Choosing the larger

ratio,  $r = \frac{108}{63.6} = 1.70$ . This may be

obtained from a slide rule or a prepared table of atomic weight ratios. Selecting the curve for a ratio of 1.7, the at. pct of 12.8 pct Ag is readily obtained. As a second example, suppose the wt pct of Ag were 70 pct in the same system. This combination requires the interchanged scales, and therefore the chart must be turned 90° clockwise. Atomic percent of  $A$  is again found by the single reading of the graph, using the same  $r = 1.7$  curve. A final example is provided by the problem of determining what weight of Fe will be contained in a given weight of  $\text{Fe}_3\text{C}$ . This is an atomic percent to weight percent conversion. Fe being the heavier component, the atomic percent to be considered is obviously 75 pct, and  $r = \frac{55.9}{12.0} = 4.65$ . Using the chart in the sideways position, the value of 93.3 wt

pct of Fe is obtained, estimating the last digit. Thus, every 6.7 g of C in the carbide are combined with 93.3 g of Fe.

It is at once recognized that the chart becomes quite inaccurate for the low and high percents and also for the high ratios. This can be overcome, if the need so warrants, by making enlarged plots of limited areas of the original whole plot. Another variation in accuracy occurs in that simple interpolation between curves becomes inaccurate as the ratio becomes large.

To summarize the conditions for which the chart is set up: (1) the percents indicated on the scales of the chart refer to the component which has the larger atomic or molecular weight or density, (2) the ratio,  $r$ , which selects the appropriate curve, is the larger ratio of weights or densities, (3) whenever a combination of percent and  $r$  falls outside the triangular area covered by the curves, the chart must be turned 90°.



# Intergranular Parting of Brass during Anneals

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Brass mills are familiar with a recurring problem which reveals itself during deformation of annealed metal as an opening up of cracks which are suggestive of a grain boundary pattern. A typical example is seen in Fig 1, which shows part of the convex surface of a cartridge brass disc which was slightly dished by the punching operation. Another illustration of the same type of defect was found in the surface of a finished cartridge case which split open on firing. These cracks, shown in Fig 2, were away from the split but indicate the presence of the type of weakness which permitted the splitting. Usually the weakness consists of separate cracks, the lengths of which are of the same order of magnitude as the grain size prior to the final anneal. Their typical appearance in a polished section is shown in Fig 3. This structure was found below the surface of a fractured tensile-specimen that had been cut from a large cartridge blank showing the defects on its convex surface.

While the pattern formed by these cracks suggests a grain boundary origin, the cracks bear no relation to the currently existing grain structure. Thus Fig 3 shows that the grains have grown up to cracks already present. Since it is doubtful that the cracks were present in the metal before the anneal, they must have formed during the anneal but prior to recrystallization. The term "fire-cracking" has been generally applied to cracking that occurs during an anneal under the influence of internal stress, but refers more specifically to obvious macroscopic cracking attributable to the presence of a low melting phase, usually lead. Since the type of cracking described above may occur when the lead content is very low, we have considered it a somewhat different phenomenon, and have called it "intergranular parting." While most examples involve cartridge brass, intergranular parting has also been observed in the fabrication of large seamless tubes from discs of both 85/15 red brass and 70/30 cupro-nickel. Similar cracking in

nickel silver was investigated by Jones and Whitehead,<sup>1</sup> who showed that it could occur during heating or cooling. That which occurred on heating they called "fire cracking," and they suggested that a transformation at about 320°C might account for its occurrence.

It was felt in this laboratory that the observed parting was probably one aspect of the general observation, first made by Rosenhain and Archbutt,<sup>2</sup> that a tendency toward intergranular fracture under tensile stress increases with increasing temperature and decreasing rate of strain. In this case the stress involved would be internal. The rate of strain would be exceedingly slow, a localized internal creep. Reference to the literature uncovered no efforts to extend the observations of Rosenhain to conditions involving only internal stress.

Accordingly, an exploratory research, sufficient in detail to satisfy us as to the probable truth of this explanation of the observed intergranular parting, was undertaken. The internal stresses present during the early stages of an anneal (prior to recrystallization) would be from two sources: residual stresses developed during deformation, and thermal stresses caused by uneven heating and expansion. Thermal stresses would vary widely according to shape, size and manner of heating, and can be considered as supplementary to residual stresses. It seemed necessary to determine the stress and temperature conditions under which parting would occur in a relatively short time, and then to establish whether or not in-

ternal stresses may persist to an extent adequate to cause parting at such temperatures. (Jones and Whitehead<sup>1</sup> and Moore and Beckinsale<sup>3</sup> made tests which indicated that stress relief required an appreciable time.) During the course of the research the desirability of studying the effect of grain size became apparent, and this factor is one of the major variables of our investigation.

## Intergranular Parting under Tension at Elevated Temperatures

In early experiments, hard 70/30 brass tensile specimens, with unknown residual stresses, held for 10 min. at applied stresses from 30,000 to 40,000 psi at temperatures from 300 to 350°C, showed no macroscopically visible cracks when unloaded and cooled, but cracks very similar in appearance to those observed in commercial practice were revealed in these specimens by pulling them to fracture. Annealed specimens, on the other hand, showed no cracks in such experiments, apparently because they deformed plastically at stresses below those necessary to cause parting in a reasonable time. Hence, the specimens used for this study were first strengthened by cold tensile elongation, giving them an unavoidable residual stress pattern (determined largely by grain size and orientation) which would, however, correspond in direction, at least, to the stress pattern obtained under stress at temperature. A variation in these residual stresses might be expected with variations in grain size, but following any given anneal the reproducibility of stress pattern should be as good as that of the grain size measurement.

With no information as to the effect of the amount of prior deformation, tests were conducted on specimens given the same cold elongation, using a stress applied at temperature which was a constant proportion of the stress required to produce this elongation. In

New York Meeting, February 1950.

TP 2717 E. Discussion of this paper (2 copies) may be sent to *Transactions AIME* before April 1, 1950. Discussion is tentatively scheduled for publication in November 1950.

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<sup>1</sup> References are at the end of the paper.





FIG 1—The convex surface of a 70/30 brass cartridge disc (6.29 in. diam  $\times$  0.525 in.). Slightly dished during punching.  $\times$  10

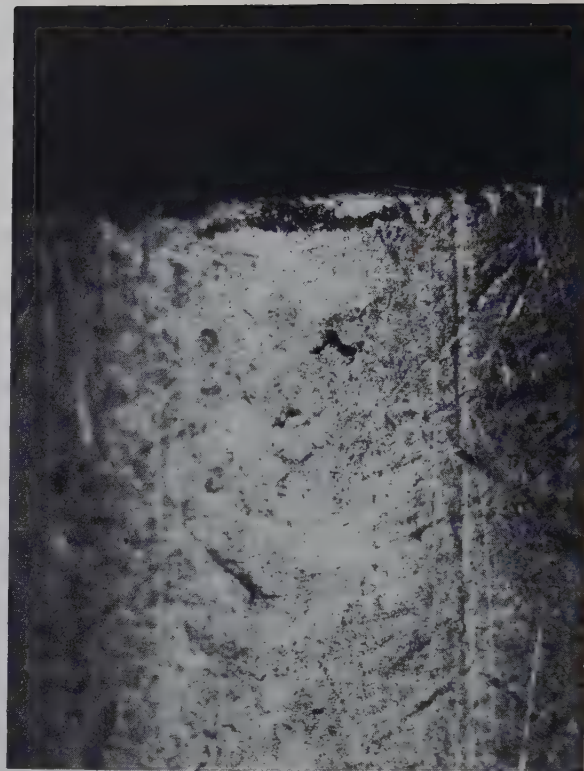


FIG 2—Cracks near head of 70/30 brass 0.270 cal. cartridge which split on firing.  $\times$  30



FIG 3—Cracks in a broken tensile specimen cut from a 70/30 brass cartridge disc ( $16\frac{1}{16}$  in. diam  $\times$   $1\frac{1}{16}$  in.) which showed surface cracks as punched.  $\times$  75

### Tests at Elevated Temperatures under Sustained Loads

Tensile specimens were machined to a  $\frac{3}{8}$  in. diam over a 2 in. ga length from  $\frac{9}{16}$  in. diam 70/30 brass rod, which had been given a final reduction of about 30 pct. The ready-to-finish grain size was 0.065 mm. Analysis showed the following composition:

	Pct
Copper.....	69.64
Zinc.....	30.33 by difference
Lead.....	0.02
Iron.....	0.01

Specimens were annealed for 1 hr at 400, 500, 600, 700 and 800°C and developed grain sizes of 0.020, 0.035, 0.1, 0.2 and 1 mm respectively. For each test, a specimen was stretched 25 pct, noting the maximum load and final diameter. It was then set in the tensile machine and brought to within  $\pm 3^\circ$  of the desired temperature by means of an electric furnace. After soaking 15–20 min. it was loaded quickly to 80 pct of its prior maximum load. This load was automatically maintained for 10 min., save in a few cases where a maximum crosshead travel of 0.01 ipm failed to compensate for creep. The extent of damage by cracking was revealed by subsequently pulling the specimen to fracture at room temperature, the maximum load and the diameter at fracture being observed. Tensile

this way the stress applied at temperature was approximately related to the yield strength of the material tested. As a result, large grained specimens were held under lower stresses at tem-

perature than the fine grained ones. Later tests determined the relative behavior of specimens having different grain sizes which were given the same stress at the same temperature.

**Table 1 . . . Intergranular Parting of 70/30 Brass Under Stress at Elevated Temperatures**  
Specimens Stretched 25 Pct before Stressing

		Stress at Temperature		Room Temperature Test to Fracture	
Temperature during Stressing	Pre-stress $\times 10^{-3}$ psi	Stress psi	Pct of Pre-stress	Tensile Strength	Reduction of Area Pct
Based on Area after Pre-Stressing					
400° Anneal—0.020 mm grain diam					
225	56.7	45,300	80	63,300	71.2
250	55.7	44,500	80	62,900	64.6
275	56.7	45,400	80	63,500	54.8
300	56.5	45,400	80	62,300	54.0
325	56.3	31,500	56	61,200	53.6
450° Anneal					
323	53.6	31,000	58	55,600	39.6
500° Anneal—0.035 mm grain diam					
225	52.3	41,800	80	60,600	68.8
250	49.3	40,200	80	58,400	61.8
275	50.0	39,900	80	56,900	47.2
287½	52.1	40,400	77.5	54,800	28.2
300	50.3	34,500	68.5	54,600	38.3
323½	50.7	31,400	62	52,400	32.2
600° Anneal—0.1 mm grain diam					
225	44.9	36,000	80	55,800	70.6
250	42.0	33,500	80	53,200	64.8
275	41.9	33,400	78.5	49,300	35.8
286*	44.8	35,850	80		5.4
300*	44.8	35,800	80		2.0
700° Anneal—0.2 mm grain diam					
225	38.8	31,100	80	51,100	61.6
250	36.9	29,500	80	50,600	69.4
275	37.5	29,900	80	48,800	49.8
277	38.8	31,100	80	47,100	36.3
280½	38.9	31,100	80	46,700	40.5
300*	40.9	32,600	80		5.7
800° Anneal—1 mm grain diam					
225	36.8	29,500	80	50,100	56.3
250	30.7	24,500	80	45,300	63.1
275	32.0	25,500	80	43,100	42.9
279*	37.2	29,700	80		8.3
300*	36.8	29,400	80		5.4

\* Broke at temperature in furnace.

strength and reduction of area values, based on the diameter of the specimen just before the final tests, were recorded.

With this procedure, tests were made on specimens representing all the grain sizes listed above at temperatures of 225, 250, 275 and 300°C. In addition, tests were made at 325°C for specimens annealed at 400, 450 and 500°C, and a few other tests were made at intermediate temperatures.

The results of these tests, as recorded in Table 1, are grouped according to the temperature of anneal, and within each group are in the order of increasing temperature of stressing. The value in the "pre-stress" column (the maximum true-stress reached with cold elongation) is an indication of the strength of the specimen under load.



**FIG 4—The variation of intergranular parting with grain size and temperature.**  
Longitudinal sections showing cracks opened up by room temperature fracturing of 70/30 brass tensile specimens annealed 1 hr at the indicated temperatures, stretched 25 pct, and stressed at 80 pct of the prior maximum stress for 10 min. at the indicated temperatures.  $\times 2\frac{1}{2}$ .

It will be noted that for anneals of 500°C, and below, it was not possible to reach 80 pct of the pre-stress at some of the temperatures. In such cases, the specimen stretched continuously at the maximum permitted loading rate of 0.01 ipm for the full 10 min. under load. As indicated, several specimens broke in the furnace before the 10 min. had elapsed. In these cases the fracture was completely intergranular and there was no evidence of local necking before fracture.

Damage from cracking is indicated by a lowering of tensile strength and of ductility as measured by reduction of area. Inspection of the data shows that for any one grain size the amount of damage increases with the temperature of stressing. There is an apparent downward trend of tensile strength and ductility with increasing grain size, and the reality of this effect will be shown in tests at constant stress which will be discussed later. Photographs of polished longitudinal sections through the fractures of most of the specimens (Fig 4)

demonstrate a correlation between ductility and cracking. Cracks, present even in the fine grained specimen tested at 225°C, increase in number with the temperature of test and with the grain size up to the 600°C anneal. The greater magnitude of each crack in the larger grained specimens overcompensates for the decreased number of cracks, and represents greater damage as measured by reduction of area.

Cracks on the surface near the fracture of specimens annealed at 600, 700 and 800°C are illustrated in Fig 5. These bear a strong resemblance to those found when intergranular parting is encountered in commercial fabrication, as illustrated in Fig 1 and 2.

The data from this series of tests are also presented graphically in Fig 6. Three variables are plotted. (A fourth, stress at temperature, is not included, but can be found by reference to Table 1.) The vertical axis indicates the reduction of area, the variation of which with the temperatures of anneal (grain size) and the temperatures of



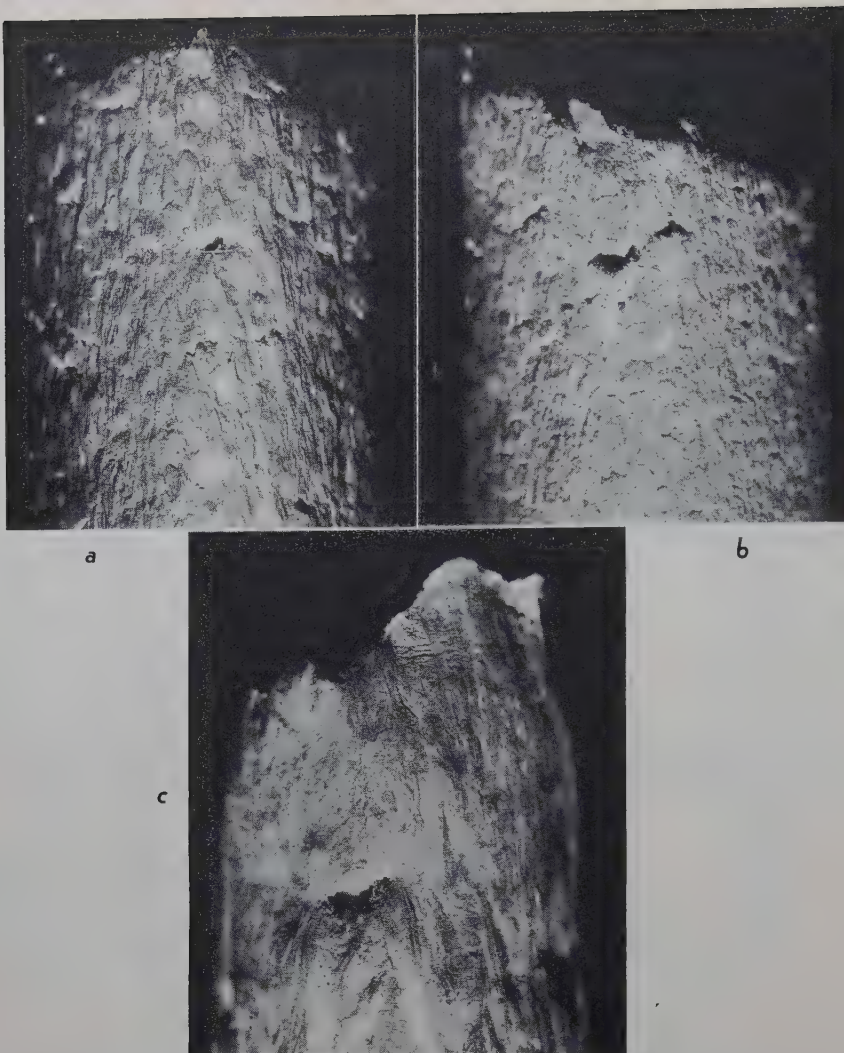


FIG 5—Intergranular parting revealed by room temperature fracturing.

Tensile specimens annealed 1 hr at indicated temperature, elongated 25 pct, and loaded for 10 min. at indicated stresses and temperatures.  $\times 10$ .

	Anneal, °C	Stress, psi	Temperature, °C
(a)	600	33,400	275
(b)	700	31,100	280
(c)	800	25,500	275

stressing constitutes a surface. Contour lines of constant temperature of stressing and constant grain size are drawn to indicate this surface. Points on the surface indicate single tests. The reduction

of area figures for specimens which broke in the furnace are included and are responsible for a sharp break in the slope of the surface. In the region of low grain size and high temperature of

Table 2 . . . Effect of Magnitude of Pre-stressing on Intergranular Parting

Grain Size Anneal	Pre-stress psi $\times 10^{-3}$	Pre-stretch Pct	Stress at Temperature, 275°C		Test to Fracture	
			psi $\times 10^{-3}$	Pct of Pre-stress	Tensile Strength psi $\times 10^{-3}$	Reduction of Area, Pct
700°C	36.3	22.5	18.2	50	50.4	71.7
	21.4	10.0	18.2	85	45.6	72.1
	35.7	20.5	21.4	60	50.8	67.8
	25.2	11.0	21.4	85.5	47.0	60.6
	35.7	20.0	25.0	70	48.7	47.0
800°C	29.1	16.5	25.0	86	47.1	48.8
	83.6	29.0	19.3	50	49.1	57.5
	22.2	12.5	18.8	85	43.4	57.2
	44.3	33.5	22.1	50	50.8	53.3
	26.0	16.5	22.1	85	43.1	49.6
	50.1	40.0	25.0	50	53.2	41.9
	29.6	20.0	25.2	85	38.9	39.1

stressing, dotted contour lines are drawn through points representing tests in which 80 pct of the prior load could not be attained.

A few tests were made which compared pairs of specimens given the same stress at temperature following different prior deformations. These were made only on specimens annealed at 700 and 800°C; specimens were stressed at about 18,500, 21,500 and 25,000 psi for 10 min. at 275°C. The results of these tests are listed in Table 2. Referring to the reduction of area figures, it will be seen that the damage is almost independent of the amount of prior deformation.\* The results confirm the expectation that the parting increases with stress. Moreover, comparison can be made between specimens of differing grain size which are given the same stress at the same temperature, and it is seen that loss of ductility from parting does increase with increasing grain size. For each pair, differing only in the amount of prior reduction, the tensile strengths are less consistent measures of damage than the reduction of area values. In each case a higher tensile strength is indicated for the higher prior deformation and, since the strength value is based on the area after deformation, this is due to work hardening in uncracked portions of the specimen. All of these specimens showed visual evidence of parting except the 700°C annealed specimen stressed at 18,200 psi.

## Discussion of Results

Since these test results involve only single specimens under each condition, there is no claim that any absolute values can be read from the table; nor does it seem necessary to try to establish absolute values, since in the practical application of this information actual stress, temperature, and grain size values are rarely known. The important points are the approximate determination of the conditions which will cause intergranular parting and the directions of the effects of the variable factors studied.

It is shown that 70/30 brass will part in less than 10 min., at grain boundaries, at temperatures below those which cause recrystallization in a reasonably short time, under the influence of

\* That this is not universally true was learned in a few comparable tests on silicon bronze. Specimens of equivalent grain size, given identical true stresses at the same temperature, were profoundly affected by the amount of prior deformation, or the concomitant variable, specimen diameter. The greater parting occurred on specimens where the true stresses were a higher percentage of the previously applied true stress.

stresses of approximately the same order as those which may be found internally. The extent of parting will increase with both increasing temperature and stress. The loss of ductility resulting from the parting increases with increasing grain size even when the number of cracks per unit volume decreases. At least for 70/30 brass, susceptibility to parting is independent of prior cold work, provided the metal has been strengthened sufficiently to support the required level of stress.

The type of test performed here differs from tensile tests at low rates of strain at elevated temperatures in that, by holding the load constant at a stress below the yield strength, the strain rate is exceedingly low. The test differs from stress-rupture and creep tests in the time allowed for stress application at temperature. However, as in these other types of test, failure occurs by parting at the grain boundaries and it is believed that the present data add to an accumulation of results<sup>2,4-12</sup> which indicate that the only conditions necessary to cause parting at grain boundaries under stress are a sufficiently high temperature and a sufficiently low rate of strain. Theoretical interpretation of this behavior is ably discussed by Zener<sup>13</sup> in terms of the viscous behavior of grain boundaries.

Experimental Study of  
Some Factors Influencing  
Relief of Stresses by  
Annealing

Having demonstrated that intergranular parting can occur under the influence of externally applied stress at elevated temperatures, there remained an investigation of the probability that adequate stresses, thermal and residual, can be present internally when metal is heating up in commercial anneals. The many unpredictable factors involved in the development of thermal stresses makes an investigation of these quite difficult. However, relaxation tests on bent strips, while limited for quantitative studies by the fact that stress formulas assume a linear stress distribution, can give at least a qualitative estimate of the rate of relief of internal residual stress and can determine the factors which influence it.

STRESS RELIEF TESTS

Relaxation tests, involving measurements of residual curvature of initially

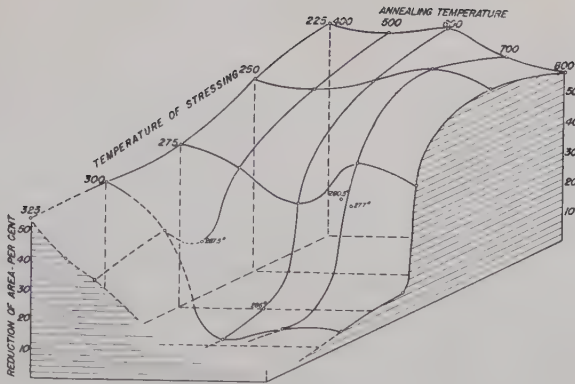


FIG 6—The effect of grain size and temperature of stressing on ductility.

Same specimens as in Fig 4. Reduction of area values based on dimensions after stressing. Temperature in °C.

flat specimens annealed while constrained against a cylindrical block, were made at four different stresses (by varying the strip thickness), for five different grain sizes, and at three temperatures in the relief annealing range. The specimens were cut from four strips of 70/30 brass fabricated from hot rolled stock having the following composition:

	Pct
Copper.....	69.81
Zinc.....	30.16 by difference
Lead.....	0.02
Iron.....	0.01

Table 3 outlines the course of fabrication for the four gauges. The ready-to-finish anneals resulted in the following grain sizes, substantially the same for all four gauges:

400°C—0.012 mm	600°C—0.115 mm
500°C—0.038 mm	700°C—0.325 mm
800°C—0.450 mm	

It will be noted that the strip was in all cases finished with a 25 pct reduction, since, as in the case of the tensile specimens stressed at elevated temperatures, a hard material was necessary to permit development of stresses of the desired magnitude. Half inch wide specimens 6 in. long were cut from the finished strip, straightened somewhat by stretching, and milled to 0.45 in. width.

A few additional tests were made at one temperature on even thinner material, in two grain sizes, which was prepared by taking some of the 0.049

in. material rolled 25 pct and processing as follows:

	Anneal 1 hr at 650°C	
	5E, 6E, 7E, 8E	5A, 6A, 7A, 8A
Cold roll to.....	0.022 in.	0.022 in.
Anneal 1 hr at.....	400°C	800°C
Cold roll 15 pct to	0.0185 in.	0.0185 in.

Samples 5A–8A had uniform grain sizes of about 0.375 mm while samples 5E–8E had an uneven grain size averaging 0.020 mm. Note that this material was finished with a reduction of only 15 pct, rather than the 25 pct used for the more extensive series above.

The stress measurements were based on calculations of outer fiber stresses in bent strips, assuming a linear distribution of stress. The amount of bend was determined from the height of the segment intercepted by a 5 in. chord. The stress required to change the curvature from that determined by a segment height  $d_a$  to that determined by  $d_b$  is

$$S_a - S_b = Et/6.25 (d_a - d_b)$$

where  $E = 16,400,000$  psi and  $t$  is the thickness. With  $d_1$  the height after holding a specimen against a 20 in. radius surface for 5 min. at room temperature,  $d_2$  the height after a subsequent 60 min. anneal of the bent specimen, and  $d_0$  the segment height for a 20 in. radius of curvature,

Table 3 . . . Preparation of Strips for Stress Relief Tests

Final Gauge.....	0.037 in.	0.049 in.	0.0615 in.	0.0735 in.
	Hot rolled stock milled to 0.500 in. Annealed 2 hr at 750°C—pickled			
Cold rolled to.....	0.200 in.	0.260 in.	0.300 in.	
Cold rolled to.....	0.100 in.	0.130 in.	0.160 in.	0.200 in.
Cold rolled to.....	0.049 in.	0.065 in.	0.0815 in.	0.098 in.
Cold rolled to.....	0.037 in.	0.049 in.	0.0615 in.	0.0735 in.
	1 piece of each annealed at 400, 500, 600, 700, 800°C (25 Pct Reduction on Finished Strip)			



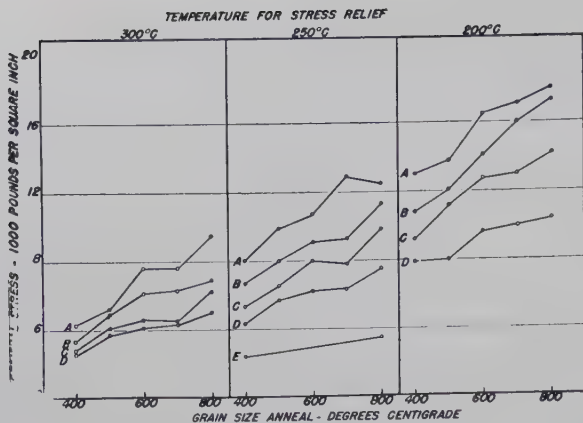


FIG 7—The effect of grain size and temperature on stress relief.

Residual stresses in bent 70/30 brass strips after 1 hr at indicated temperatures when initially stressed to about: A. 26,000 psi B. 22,000 psi C. 19,000 psi D. 15,000 psi E. 7,000 psi

$S_0 - S_1$  is the applied stress before relief annealing.

$S_2 - S_1$  is the stress relieved by the anneal

$S_0 - S_2$  is the residual stress which returns the specimen to its final position after the anneal.

The assumption of linearity of stress distribution is known to be inaccurate in two ways: 1. Any plastic flow occurring during the 5 min. bending at room temperature leaves a residual stress which decreases the applied stress below that calculated by an undetermined amount.<sup>14</sup> 2. The stress relief, occurring disproportionately in the outer fibers, destroys any linearity of stress distribution and calculations based on the final curvature give an outer fiber stress higher than that

actually present.

Nevertheless, since the actual stresses must be related to the measured ones, the technique is useful for determining the relative effect of variable factors affecting stress relief, such as temperature, time, grain size and the magnitude of applied stress.

The 20 in. radius bend was obtained by the use of a steel block with a surface having this curvature against which the specimens were free to slide in expanding. Due to the fact that the heavier gauges took on a permanent set at room temperature the calculated applied stresses were 15,000, 19,000, 22,000 and 26,000 psi rather than the intended (assuming flat specimens) 15,000, 20,000 25,000 and 30,000 psi. In the anneals, 60 min. was allowed for the assembly to reach temperature.

Table 4 . . . Stress Relief in 70/30 Brass

Gauge Inch Av.	Grain Size mm	Applied Stress			Stress Relieved Thermally						Residual Stress		
		$S_0 - S_1$ psi $\times 10^{-3}$			$S_2 - S_1$ psi $\times 10^{-3}$			$\frac{S_2 - S_1}{S_0 - S_1} \times 100$ per cent			$S_0 - S_2$ psi $\times 10^{-3}$		
		200°C	250°C	300°C	200°C	250°C	300°C	200°C	250°C	300°C	200°C	250°C	300°C
0.0735	0.450	26.3	25.6*	26.7	8.4	13.0*	17.3	31.9	50.8*	64.8	18.0	12.5*	9.5
	0.325	26.4	28.4	25.8	9.3	15.5	18.2	35.2	54.6	70.5	17.1	12.9	7.6
	0.115	26.8	24.3	26.2	10.3	13.7	18.5	38.4	56.4	70.6	16.5	10.7	7.6
	0.038	25.7	27.4	23.9	11.9	17.5	18.7	46.3	63.9	78.2	13.8	9.9	5.2
	0.012	27.5	26.4*	26.8	14.4	18.3*	22.4	52.4	69.3*	83.6	13.0	8.0*	4.3
0.0615	0.450	24.9	23.6	21.6	7.6	12.2	14.8	30.5	51.7	68.5	17.3	11.3	6.9
	0.325	24.6	21.6	21.6	8.6	12.3	15.3	35.0	56.9	70.8	16.0	9.3	6.3
	0.115	23.1	23.2	21.3	8.9	14.1	15.2	38.5	60.8	71.4	14.1	9.1	6.1
	0.038	22.1	21.6	20.3	10.0	13.6	15.5	45.2	63.0	76.4	12.1	8.0	4.9
	0.012	22.2	22.9	20.2	11.4	16.2	16.9	51.4	70.7	83.7	10.8	6.7	3.3
0.0495	0.450	19.8	19.5	18.7	5.7	9.7	12.5	28.8	49.7	66.8	14.2	9.8	6.2
	0.325	19.2	18.7	17.6	6.1	10.9	13.1	31.8	58.3	74.4	13.0	7.8	4.5
	0.115	20.1	20.0	19.1	7.4	12.1	14.5	36.8	60.5	75.9	12.7	8.0	4.6
	0.038	18.2	18.6	18.8	8.0	12.1	14.8	41.7	65.1	78.7	11.2	6.5	4.1
	0.012	18.5	17.4	17.7	9.3	12.2	14.9	50.3	70.1	84.2	9.2	5.3	2.8
0.0375	0.450	14.7	15.0	14.7	4.3	7.5	9.6	29.2	50.0	65.3	10.4	7.5	5.0
	0.325	14.9	14.2	14.6	4.9	7.9	10.3	32.9	55.6	70.5	10.0	6.3	4.3
	0.115	15.3	14.6	14.5	5.7	8.3	10.4	37.2	56.8	71.7	9.6	6.2	4.1
	0.038	14.9	15.2	15.3	7.0	9.6	11.6	47.0	63.2	75.8	8.0	5.7	3.7
	0.012	15.4	14.7	14.8	7.5	10.4	12.2	48.7	70.7	82.4	7.9	4.3	2.5
0.0185	0.375		7.4			3.9			52.7			3.5	
	0.020		6.7			4.3			64.2			2.4	

\* Tests used for comparison with tests on specimens having smaller gauge. See p. 1001.

## EFFECT OF TEMPERATURE, GRAIN SIZE AND THE MAGNITUDE OF APPLIED STRESS ON STRESS RELIEF

Specimens for each grain size in the four heavier gauges were relief annealed for 1 hr at 200, 250 and 300°C. The 0.0185 in. strips were relief annealed only at 250°C. Data as determined from measurements before and after the anneal are given in Table 4 and plotted in Fig 7 and 8. The per cent stress relieved thermally based on the applied stress is also included. These data illustrate the expected lower residual stresses resulting from higher relief annealing temperatures. It is clear from the consistency of behavior for all gauges and temperatures that specimens with finer grain sizes have less residual stress than those with coarse grains. Moreover, the amount of residual stress increases with applied stress, the curves in Fig 8 demonstrating about the same level of per cent stress relief at any one temperature, regardless of the magnitude of the applied stress.

With reference to the 0.0185 in. specimens stressed to only 7,000 psi (Table 4 and Fig 7), it will be seen that, at least at 250°C, if there is any minimum stress below which there is no relief annealing it must be very low. It is interesting to note that even at low stresses the per cent stress relief is not affected by applied stress. The fine grained (0.020 mm) thin specimen shows a per cent stress relief between the averages for 0.012 mm (400°C) and 0.038 mm (500°C) thicker specimens, and the coarse grained (0.375 mm) thin specimen a value between those for 0.325 mm (700°C) and 0.450 mm (800°C) thicker specimens.

Even under conditions of least stress relief (that is, with large grained specimens), the magnitudes of residual stresses at temperature, after 1 hour of relief annealing, are well below those required for intergranular parting at any of the temperatures employed. Since it seemed probable that the stresses remaining after shorter times at temperature would be higher, the change of residual stress with time was tested for one applied stress on both fine and coarse grained specimens.

## EFFECT OF TIME ON STRESS RELIEF

For studying the effect of time on relief annealing, the steel block was unsatisfactory since it required an hour to reach temperature. A thin walled

tube, 10.1 in. od  $\times$  0.063 in. wall, which required only 6 min. to reach temperature, was employed. The smaller radius called for thinner specimens for equivalent stresses, and the 0.0185 in. thick material designated by 5A-8A and 5E-8E was sheared to size for these tests. Strip having this gauge should develop outer fiber stresses of 30,000 psi when bent to the 5.05 in. radius of the tube support. However, as a result of initial curvature and permanent set at room temperature, the applied stresses were of the order of magnitude of 25,000-29,000 psi.

One specimen for each grain size was held to the tube with wire. Again  $d_1$  after 5 min. at room temperature, and  $d_2$  after the anneal, were measured. Allowing 6 min. to reach temperature, the specimens were annealed for 0, 3 and 6 min. at 200, 250 and 300°C. An additional test was made for 60 min. at 250°C.

Data corresponding to those assembled for specimens bent to the 20 in. radius are recorded for these specimens in Table 5. The effects of grain size and temperature of relief annealing are again evident.

The effect of time is presented graphically in Fig 9. Since tests for 60 min. were made only at 250°C, the data for the points at 60 min. for the 200 and 300°C curves are taken from Table 4 and are indicated by filled circles. These results were obtained with thicker and harder specimens bent to a larger radius of curvature, and justification for their inclusion can be made by comparing the stress relief in thick and thin specimens under equivalent stresses at 250°C for 60 min. For instance, data for specimens 6A60 and 6E60 may be compared with the two starred specimens in Table 4. By bending to different radii, these 4 specimens were given about the same applied stress and all were relief annealed for 1 hr at 250°C.

Specimen	Gauge Inch	Grain Size mm	Radius Inch	Applied Stress psi	Per Cent Stress Relief	Retained Stress psi
1	0.0735	0.450	20	25,600	50.8	12,500
2		0.012	20	26,400	69.3	8,000
3(6A60)		0.375	5	24,800	50.0	12,400
4(6E60)	0.0185	0.020	5	28,300	66.8	9,300

Specimens 1 and 3 show almost identical stress relief and the difference between 2 and 4 may be due to the effect of their difference in grain size. It is evident that with a constant applied stress specimen thickness may vary widely without greatly influencing the extent of stress relief as tested by bend-

Table 5 . . . Effect of Time on Stress Relief (70/30 Brass—0.0185 in. gauge—5 in radius)							
Specimen	Grain size mm	Applied Stress $S_0 - S_1$ psi	Relief Annealing Temperature °C	Time at Temperature min.	Stress Relieved Thermally		Residual Stress $S_0 - S_2$ psi
					$S_2 - S_1$ psi	$\frac{S_2 - S_1}{S_0 - S_1} \times 100$ Pct	
7A0	0.375	26,000	200	0	5,300	20.4	20,700
7A3	0.375	26,800	200	3	6,600	24.6	20,200
7A5	0.375	26,900	200	6	6,700	24.9	20,200
7E0	0.020	28,400	200	0	8,600	30.3	19,800
7E3	0.020	28,400	200	3	9,900	34.9	18,500
7E5	0.020	26,600	200	6	9,700	36.5	16,900
6A0	0.375	26,800	250	0	7,900	29.5	19,000
6A3	0.375	28,000	250	3	10,200	36.4	17,800
6A5	0.375	26,600	250	6	10,300	38.7	16,300
6A60	0.375	24,800	250	60	12,400	50.0	12,400
6E0	0.020	27,200	250	0	12,500	46.0	14,700
6E3	0.020	29,200	250	3	15,600	53.4	13,500
6E5	0.020	27,600	250	6	15,600	56.5	12,000
6E60	0.020	28,300	250	60	18,900	66.8	9,300
5A0	0.375	26,800	300	0	13,300	49.6	13,400
5A3	0.375	27,000	300	3	16,100	59.6	10,900
5A5	0.375	27,200	300	6	16,000	58.8	11,200
5E0	0.020	28,200	300	0	18,600	66.0	9,600
5E3	0.020	29,600	300	3	21,500	72.6	8,200
5E5	0.020	29,700	300	6	21,900	73.7	7,800

ing strips. As seen in Table 5 and Fig 9, the values at "zero" time show that there has been a rapid initial decrease in residual stress (from an applied stress of 25,000-29,000 psi) during the 6 min. heating up period to each of the temperatures, and this is greater as the temperature increases and the grain size decreases. However, this rapid decrease does not constitute the whole of the stress relief and residual stresses remain after 10 min. in the furnace (4 additional min.) which are appreciably higher than the stresses remaining in the earlier tests after 60 min. The curves suggest that, after the initial rapid stress relief, the rate of stress relief is independent of grain size and temperature.

DISCUSSION OF RESULTS

The data show that the amount of stress relief increases with time and temperature. This behavior would be expected of reactions involving atomic motions which require an activation

of the fact that stress relaxation and creep are fundamentally alike<sup>15</sup> and the creep rate of brass varies inversely with grain size.<sup>16</sup> The experiments were not complete enough to establish whether, given sufficient time, specimens would reach the same residual stress for a given temperature regardless of initial stress; but the results suggest that this would not be true. Thus, if there were a residual stress determined only by temperature after a very long time, it could be inferred from the curves that for some of the more highly stressed samples (for example, 28,000 psi—250°C), the final residual stress could not be much below 10,000 psi. However, a sample stressed initially at 7,000 psi drops in 60 min. to 3,500 psi. More extensive studies of Moore and Beckinsale<sup>3</sup> on the effect of time show that, except for much harder strip in which stress was entirely relieved (presumably by recrystallization) at 250-300°C, a dependence of residual stress on initial stress is maintained after the time curves become horizontal.

Thermodynamic considerations would suggest that, if the metal were homogeneous, uniform stress in an elastically strained specimen would tend to approach, with time, a residual stress characteristic of the temperature and independent of the magnitude of the initial stress. The experimental evidence that the residual stress is a definite function of the applied stress can be understood by analysis of the inhomogeneities present and of the stress distribution. Considering, first, the lack of homogeneity of stress in a polycrystalline metal, the total stress can be considered as a composite of



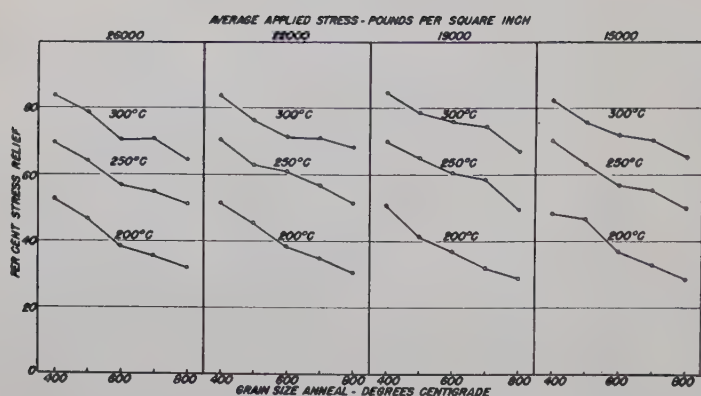


FIG 8—The constancy of per cent stress relief with varying initial applied stress. The same data as in Fig 7.

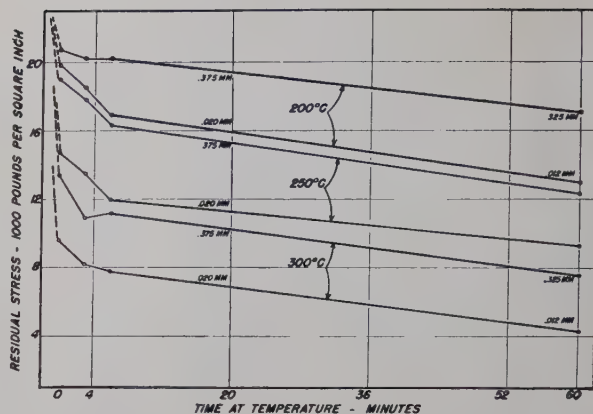


FIG 9—Rate of stress relief as affected by grain size and temperature.

Initial applied stress: 25–29,000 psi.

Datum points are residual stresses after 0, 3, 6, and 60 min. at temperature.

microstresses which vary in magnitude because of the anisotropy of elastic modulus. If each local site of microstress were considered homogeneous, overall stress relief at a given temperature would consist of a reduction of all higher stresses to a level characteristic of the temperature, and residual stress would be a composite of all the unchanged lower stresses and an increased number of stresses at the maximum level characteristic of the temperature.

Increasing the applied stress increases each microstress proportionally causing the total stress to consist increasingly of microstresses higher than the stress to which a given temperature will relax them. Thus there is more stress relief in specimens having a greater initial stress. However, the residual stress will also be greater because the extent to which the total residual stress consists of the highest stresses possible at the temperature of annealing will increase with initial stress.

With respect to the stress distribution in an elastically bent specimen, there is again a range of stress, in this case increasing linearly from the neutral axis to the surface. The same argument applies and stress relief results in a reduction of all higher stresses to a level characteristic of the time and temperature. A nonlinear stress distribution results, but the total stress will be higher the higher the initially applied stress.

Because of the uncertainty introduced by the fact that the calculated residual stresses indicate values which are higher than the actual residual stresses in the outer fibers by an undeterminable amount, it is not safe to estimate from the experimental results the magnitude of residual stress which might be found within a metal

after any particular time in an annealing furnace. The results do show, however, that whatever the magnitude of stress remaining after a given treatment of brass in a given condition it will be greater after shorter times, at lower temperatures, with larger grain sizes and with higher initial internal stresses.

### Intergranular Parting During Anneals

It has been shown that "intergranular parting," a type of failure occasionally observed in commercial fabrication of 70/30 brass, has a structural appearance which can be duplicated by holding tensile specimens at a constant stress at elevated temperatures. The most probable time in commercial fabrication that the metal is under appreciable stress at an elevated temperature is during the heating up period of an anneal. That parting occurs at this time is shown by tests which indicate that annealed brass will not support stresses adequate to cause parting in a short time (at least when they are only uniaxial), and by the observation in commercial failures that recrystallization occurs after cracking. (Some examples are known in which thermal stresses during rapid spray cooling of annealed metal appear to have caused intergranular parting).

In investigations as to whether internal stresses can be responsible for cracking, qualitative measurements of the residual stresses still present after a few minutes at the temperatures favorable to parting, at least with initial stresses up to 28,000 psi, have shown that such residual stresses may be fairly high but probably not high enough to cause the parting. Higher

residual stresses may be present when the initial internal stresses are greater. Total stresses adequate to cause parting can be accounted for, by assuming that, when this type of failure does occur, the residual stresses are supplemented by thermal stresses from uneven expansion.

The tests have shown the importance of grain size, since both the tendency toward intergranular parting and the tendency toward slow relief of stress are increased with increasing grain size.

The comparative rarity of occurrence of intergranular parting suggests that extreme variations of some of the factors contributing to it, or a fortuitous combination of several of them, must be necessary. Very high internal stresses can be sustained by heavily worked brass; the extent and persistence of thermal stresses can vary according to the type of heating and furnace construction; the rate of heating can be very rapid, minimizing stress relief and increasing thermal stresses; grain sizes may be particularly large especially in heavy gauge material, which is more susceptible to uneven thermal expansion and in which, as a matter of fact, most examples of intergranular parting are found.

Corroboration of the conclusions drawn from this work can be found in practical solutions to commercial fabrication problems, based on this explanation for the cause of intergranular parting. For example, in the early stages of fabrication of large seamless 85/15 brass tubing, intergranular parting at the shoulders of large cups can be caused, or eliminated, depending upon the position of the cups during annealing. With the open face up, the inside surface of the cup is heated most rapidly and

transfers tensile stresses to the outside. These add to the internal stresses already present at the shoulder, and frequently cause cracking. If the cup is placed bottom up on the pan, the outside surface is heated first, and the residual tensile stress at the shoulder tends to be relieved by the thermal expansion of the metal so that no cracking occurs. This evidence suggests that parting can be caused solely by the effect of stress at an elevated temperature. The possibility that compositional or precipitation effects may be contributory is not denied, but they alone are not adequate to cause parting.

The tendency toward intergranular parting can be minimized by: 1. Minimizing internal stress left by cold working. 2. Keeping the grain size down. 3. Providing for uniform heating. 4. Heating slowly enough to permit some stress relief before cracking temperatures are reached. 5. Placing the work in the furnace in such fashion that surfaces known to be internally stressed in tension reach temperature first.

The finding of cracks in the heads of 3 in., 50 caliber cartridge cases made from hot rolled 70/30 brass has recently been reported by Arnold.<sup>17</sup> The report lacks sufficient detail to indicate whether or not the factors discussed in this paper are responsible, but it is suggested that the results of this research should be considered in that connection.

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## A Proposed Microbending Mechanism of Plastic Deformation\*

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The distortion of crystal structures referred to as "biege gleitung",<sup>1</sup> "local curvature"<sup>2-4</sup> and "deformation bands"<sup>5,6</sup> is believed to be an unavoidable characteristic of deformed single crystals. The presence of asterism on Laue photograms of Al single crystals either deformed by pure shear<sup>7</sup> or extended in a condition relatively free from the grip effect<sup>2</sup> indicates strong evidence substantiating this point of view. The type of localized bending described in connection with the bending test of Al single crystals by Yen and Hibbard<sup>8</sup> could conceivably exist either microscopically or submicroscopically in all types of deformation. The purpose of this note is to extend this idea to the postulate of a hypothetical "microbending site," as applied to the mechanism of slip in a general sense.

The mosaic structure, as postulated by Darwin,<sup>9</sup> is that crystals were actually composed of small blocks which have a linear dimension  $10^{-4}$  to  $10^{-6}$  cm on the edge and deviate from each other by an angle of about one-tenth of a degree. The following discussion is based on the assumption

that when slip is propagated from one mosaic block to the adjacent one, bending of the lattice at the region of their boundary is produced in order to maintain the continuity of the gliding process along a certain slip plane.

Consider a two-dimensional diagram of two ideal mosaic blocks on the cross-section perpendicular to the slip plane and containing the active direction of slip. When an external force is applied, the localized bending or "microbending site," would be generated at the region as illustrated in Fig 1A at point B.

A schematic diagram of a "microbending site," generated at a mosaic boundary, is illustrated in Fig 1B. It may be seen that once the curvature

of the bending site reaches sufficient magnitude, the interaction force encountered by pairs of atoms symmetrical with respect to the center of the bending is equal but opposite in sign. Consequently, the overall energy required to displace the atoms near the center is approximately zero.<sup>10-12</sup>

Based upon the finding that the misalignment between two mosaics is 0.1 degree,<sup>9</sup> the assumption is made that "microbending sites" may possibly exist in the region where one plane will have one more row of atoms than an adjacent plane. It is probable that the origin of the bending site will be where the distance between two mosaic boundaries is "2d" as shown in Fig 1A at point B, where "d" is the interatomic distance. Thus, the distance between two "microbending sites" is in the range of about 600 interplanar spacings. In the case of aluminum, the spacing between two octahedral planes is about 2.33 Å. The distance between two bending sites will be approximately  $2.5 \times 10^{-5}$  cm. It is interesting to note that according to Woods<sup>13</sup> these values approach closely the size of the domains of a cold-worked metal whose minimum

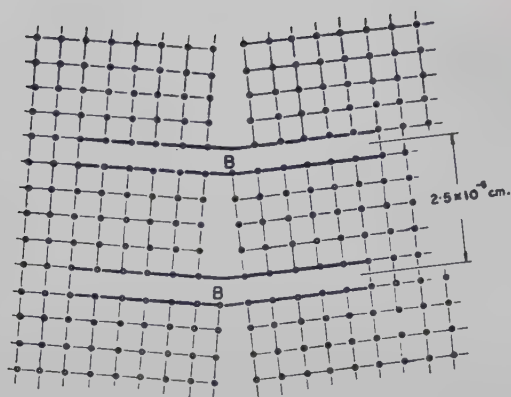
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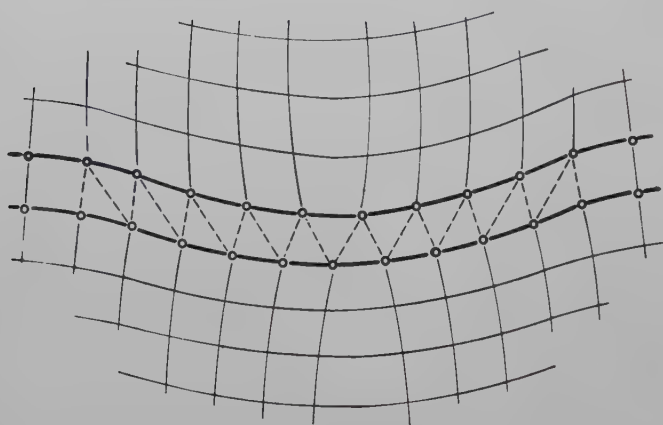
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<sup>1</sup> References are at the end of the paper.





(A)



(B)

FIG 1—Schematic diagrams showing "microbending sites."

dimension should be about  $10^{-5}$  cm.

Since different mosaic blocks in a crystal may bear various angular relationships with neighboring ones, the "microbending sites" could be classified into two types. The one with the concave side upward as shown in Fig 1B is designated as the concave type and the other with the convex side upward as the convex type. A greater interference would be exerted between a concave and a convex bending site, one immediately above the other, than two similar ones, because of the distortion produced in the adjacent lattice. The interaction force between two "microbending sites" of opposite curvature may be either attractive or repulsive, depending upon their relative position to each other. For instance, if two "microbending sites" have both concave sides opposite to each other, the tensile strain in the lattice between them would have a tendency to pull them together. If two bending sites have both convex sides opposite to each other, the interaction force becomes a repulsive one.

However, both of these two arrangements can be considered to be responsible for the strain-hardening effect in plastically deformed crystals.

One of the essential features of the mechanism proposed here is that the slip process is based upon the idea that slip starts at those planes where the location of "microbending sites" happens to be close enough to form a sub-microscopically wavy layer which is composed of a number of curved surfaces each having a low resistance to shear. As soon as the external force reaches this magnitude, slip takes place along the slip direction in the manner of a sequence of local processes. The time required from the generation of the "microbending site" to the commencement of slip appears to be the same as the incubation period in the nucleation theory recently proposed.<sup>14,15</sup> It may be seen that once slip commences, the distortion produced in the adjacent lattice may destroy the regular pattern of the "microbending sites." This results in the phenomenon of strain-hardening

which tends to prevent further gliding along the same plane. If, however, the external stress increases sufficiently to overcome the barriers between "microbending sites" which have relatively large misalignment at the ends, new slip markings between the old ones are produced.

In Fig. 1B only a two-dimensional "microbending site" is illustrated. Since this may be out of registry along the direction perpendicular to the paper, a type of distortion by bending about an axis parallel to the paper would be required to provide the continuity of the slip process. If, however, a second set of slip planes becomes active, it is conceivable that strain-hardening of latent slip plane would always exceed that of the operative ones in order to pass through the highly distorted lattice and to initiate slip.

In summary, "microbending sites" may be described as atomistically dislocated local curvatures which act as nuclei for the slip process. Thus, the mechanism for plastic deformation in crystals could be interpreted in accordance with the theory of local curvature,<sup>1-4</sup> the theory of dislocation<sup>10-12</sup> and the theory of nucleation.<sup>14,15</sup>

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# The Low Temperature Properties of Tin-antimony and Tin-cadmium Alloys\*

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## Introduction and Literature Survey

This is the second in a series of papers coming from this laboratory on the correlation of the low temperature tensile properties of tin-binary alloys with microstructure. These engineering data and those of the previous paper<sup>1</sup> were obtained as the initial part of a long-range program to determine the changes in the fundamental mechanical properties accompanying the beta to alpha phase change in tin and its binary alloys below 13.2°C. Since it appears now that the more fundamental studies will be delayed,\* and since no summary of the low temperature properties of these tin-base white bearing alloys was found in the literature, it was decided to publish all the engineering data thus far obtained. Accordingly, the tensile properties of thirteen binary alloys containing from 0.1 to 60 pct cadmium and 0.1 to 10.43 pct antimony measured at six temperatures from -196 to +23°C are presented here and their variations rationalized in terms of microstructure.

A literature search showed that these systems had not been investigated at low temperatures, although both the tin-cadmium and tin-antimony<sup>2,3,4,5</sup>

and the tin-rich corner of the ternary system<sup>6,7</sup> have been studied at room temperature. Hanson and Pell-Walpole<sup>5</sup> and Homer and Plummer<sup>4</sup> determined the tensile properties of alloys containing from 0 to 10 pct cadmium. The same authors<sup>3</sup> determined the

tensile strength and elongation in the tin-antimony system from 2 to 18 pct antimony at room temperature while Pell-Walpole et al.<sup>6,7</sup> studied the properties of high tin and tin-antimony-cadmium alloys. Since there were many variables besides composition in these tests, it was difficult to make a valid comparison with the room temperature data of the present study, but in general the tensile strength and ductility values are in reasonable agreement.

Pell-Walpole<sup>2</sup> also determined the effect of grain size on the tensile strength of pure tin and 1 pct alloys of both antimony and cadmium. The true maximum stress was found to decrease exponentially as the number of grains in the cross-section increased. This effect of grain size was strikingly revealed in the present work, as indicated in a later section.

Hanson and Pell-Walpole<sup>8</sup> placed the solid solubility limit of antimony in tin at 3.5 pct at 20°C, increasing to 4 pct at 190°C, and then sharply increasing to 10.3 pct at 246°C (the latter being a peritectic point). Their results are in disagreement with those of previous investigators,<sup>9</sup> and the discrepancy seems to lie in the micrographic observation of the second phase

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<sup>1</sup> References are at the end of the paper.



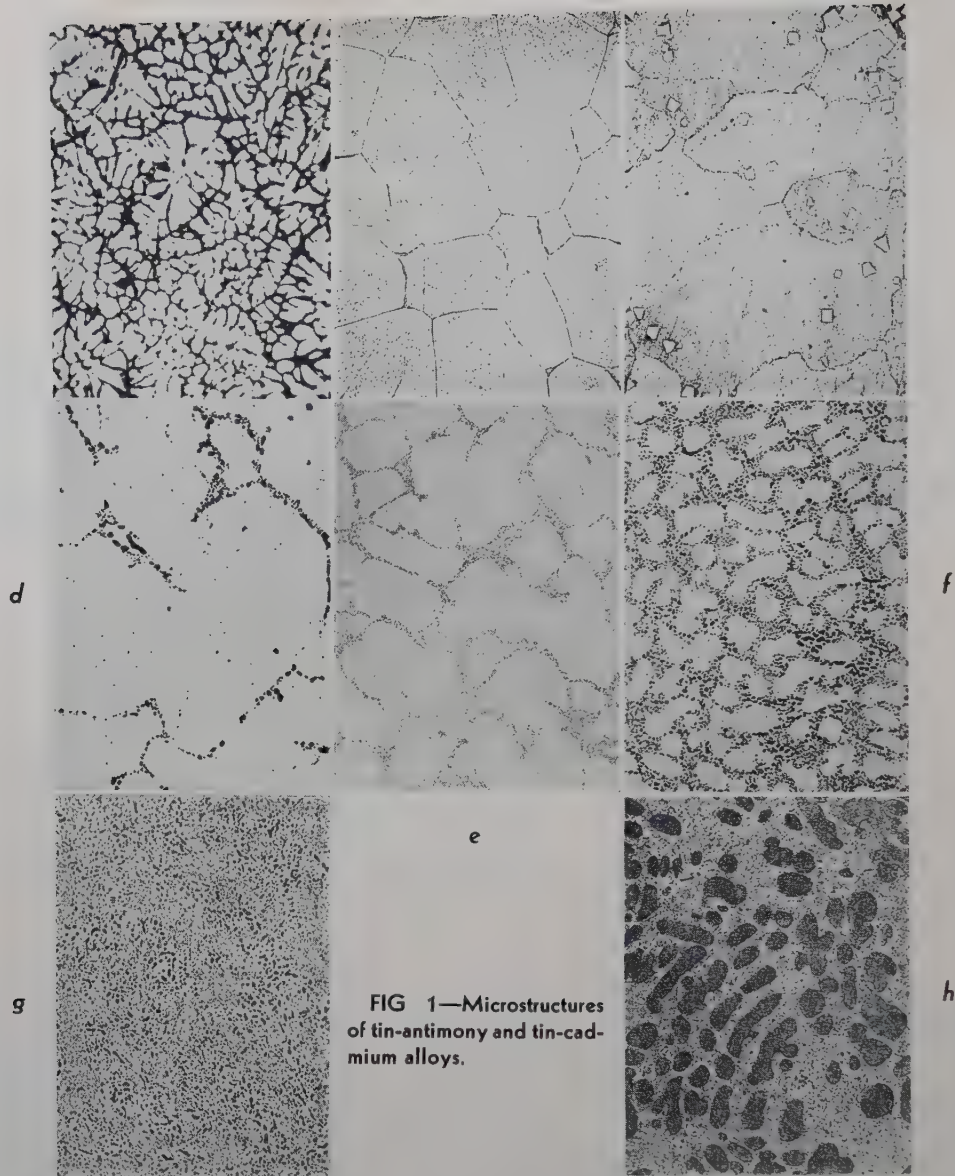


FIG 1—Microstructures of tin-antimony and tin-cadmium alloys.

*a* 3.38 pct Sb, as cast; *b* 3.38 pct Sb, homogenized 150 hr at 190°C; *c* 10.43 pct Sb, homogenized; *d* 2.65 pct Cd, homogenized; *e* 10 pct Cd, homogenized; *f* 20 pct Cd, homogenized; *g* 33 pct Cd, homogenized; *h* 60 pct Cd, homogenized. Etched in 8 parts glycerine, 1 part acetic acid, 1 part nitric. 100 X. Reduced one third in reproduction.

SbSn. This is ordinarily seen as large cuboids first appearing at about 9 pct antimony, but Hanson and Pell-Walpole showed that it also appears as a fine precipitate, preferentially along the grain boundaries, in compositions as low as 3.5 pct. They employed thermal analysis, resistance measurements and micrographic methods, and the results seem quite conclusive.

The same authors,<sup>5,9,10</sup> investigating the tin-cadmium system, found the room temperature solid solubility limit at 1.0 pct and this limit remained essentially constant to 133°C, the temperature of the eutectoid transformation.

In the present investigation, the equilibrium diagrams of Hanson and Pell-Walpole for both systems have been accepted as conclusive, since they are the most recent and the most

comprehensive. Moreover, the limited metallographic study carried out here for purposes of correlating microstructure and tensile properties, confirmed their findings.

## Materials and Methods

### PURITY AND PREPARATION OF ALLOYS

The tin was the same grade (99.9965 pct pure) used in the early investigation.<sup>1</sup> The antimony metal was Mallinckrodt reagent quality in crushed granular form and was 99.84 pct pure. Cadmium metal of 99.97 pct purity was obtained in the form of  $\frac{3}{8}$  in. rods from the American Smelting and Refining Co.

Using the melting procedure previ-

ously described,<sup>1</sup> tin-cadmium alloys containing 0.092, 0.26, 0.90, 2.65, 10.0, 20.0, 33.0, 60.0 pct cadmium, and antimony alloys containing 0.0, 0.31, 1.11, 3.38 and 10.43 pct antimony were melted and cast tensile bars, 0.25 in. in diam and 1 in. in gauge length, were prepared. Chemical analyses were made on all compositions, antimony and cadmium contents being determined spectrographically, while wet analytical methods were used for the higher values. Spot checks on the finished test specimens showed that iron, in amounts of 0.005 pct, was the greatest impurity.

### HEAT TREATMENT AND MICROSTRUCTURE

Heat treatments to provide a variety of microstructures were possible with

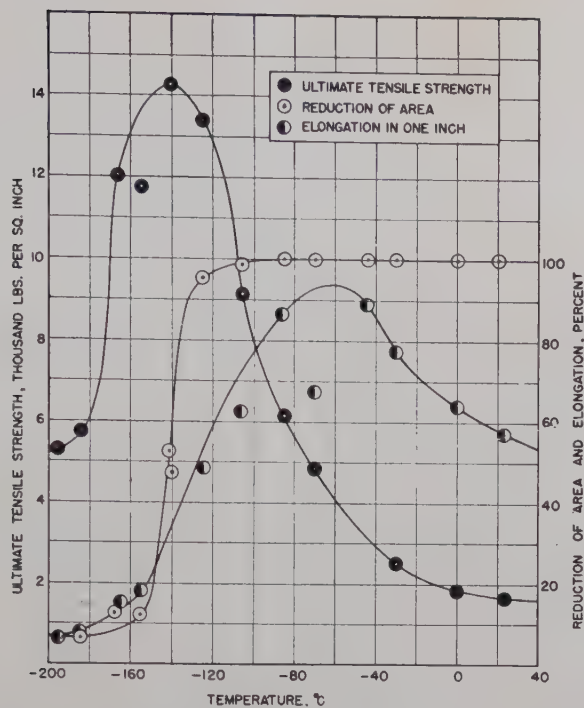


FIG 2—The effect of temperature on the tensile properties of pure tin.

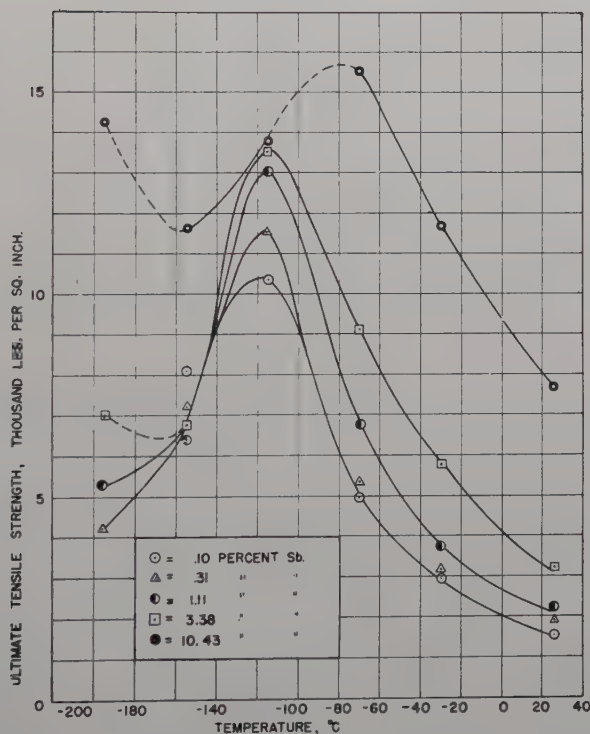


FIG 3—The tensile strength from  $-195^{\circ}\text{C}$  to  $23^{\circ}\text{C}$  of tin-antimony alloys containing 0.1 to 10.43 pct antimony.

these alloys, but a structure stable at room temperature was selected as most desirable for these studies. Consequently, the bars were annealed at a temperature such that, in cooling down to room temperature, no phase change

and only slight changes in solubility would occur. Homogenizing anneals of 150 hr at  $190^{\circ}\text{C}$  for the antimony alloys and 350 hr at  $120^{\circ}\text{C}$  for the cadmium alloys, while immersed in silicone oil, fulfilled these conditions. For con-

venience, all compositions of the respective systems were heat treated at the same temperature, since this did not violate the above imposed conditions. After the homogenization, the bars were slowly cooled in the furnace



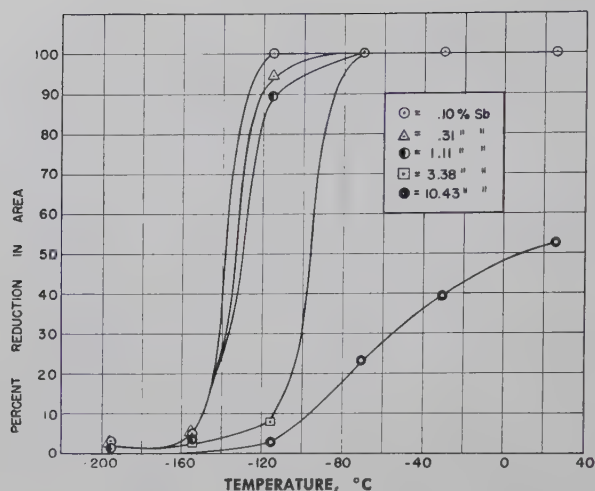


FIG 4—The effect of various temperatures on the reduction in area for tin-antimony alloys containing from 0.1 to 10.43 pct antimony.

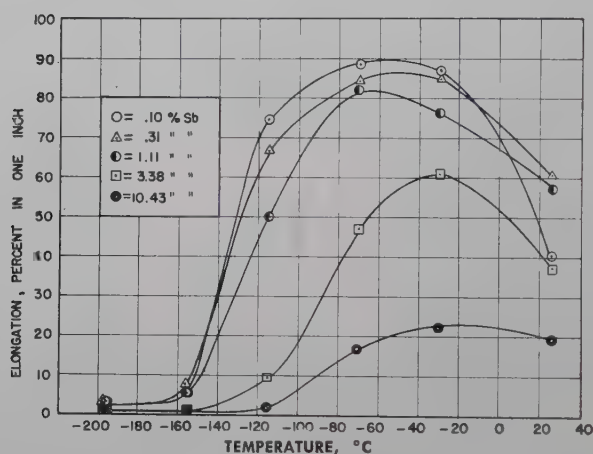


FIG 5—The percent elongation in one inch from  $-195^{\circ}\text{C}$  to  $23^{\circ}\text{C}$  for tin-antimony alloys containing from 0.1 to 10.43 pct antimony.

to room temperature.

Fig 1a shows the typical cast structure with pronounced coring obtained in the 3.38 pct antimony alloy. The structure after homogenization at 150 hr at  $190^{\circ}\text{C}$  is shown in Fig 1b. Attention is called to the definite grain boundary constituent appearing in Fig 1b. This evidence substantiates the findings of Hanson and Pell-Walpole who placed the solubility limit at 3.5 pct. These authors noted a definite tendency for the second phase to appear at grain boundaries, and Fig 1c, representing an alloy of 10.43 pct antimony, clearly shows the presence of the compound  $\text{SbSn}$  in the form of cubic crystals in the grain boundaries.

It should be noted here that a more complete metallographic investigation would have been desirable to supplement existing knowledge concerning solid-solubility limits, distribution of

second phases, and others, but the limited studies reported here were considered sufficient for purposes of this research. Fig 1d shows the microstructure of the 2.65 pct cadmium alloy in the homogenized condition. The eutectic structure is evident in Fig 1d, and it is clear that the solubility limit (1 pct) has been exceeded. This alloy undergoes a peritectic reaction at  $223^{\circ}\text{C}$ , and the resulting beta structure is stable down to  $133^{\circ}\text{C}$ . However, this reaction is easily suppressed by rapid cooling,<sup>10</sup> so that the eutectic of Fig 1d is believed to be present in nearly the equilibrium amount. Special attention is called to the dispersion of fine material throughout the grains in this 2.65 pct cadmium alloy. Whether this represents a small amount of the now metastable  $\beta$  phase, or whether the particles are  $\gamma$  which has transformed *in situ* from the  $\beta$  and perhaps slightly

agglomerated, is an interesting question and one which could not be further investigated here. The authors are inclined to the latter opinion, since the long homogenizing treatment (350 hr) is believed to represent a close approach to structural equilibrium.

Fig 1e, f, g and h show typical microstructures of the 10, 20, 33 and 60 pct cadmium alloys. Before discussing the individual structures, it will be noted that in all four compositions, the  $\alpha$  or continuous phase is much finer grained than in the lower cadmium alloys. As will be explained in the discussion of results, and mentioned later in this section, this is an important factor to be considered in the interpretation of the tensile data.

The structure of the 10 pct cadmium alloy (Fig 1e) is seen to be two phase with the  $\gamma$  phase forming an almost continuous grain boundary envelope

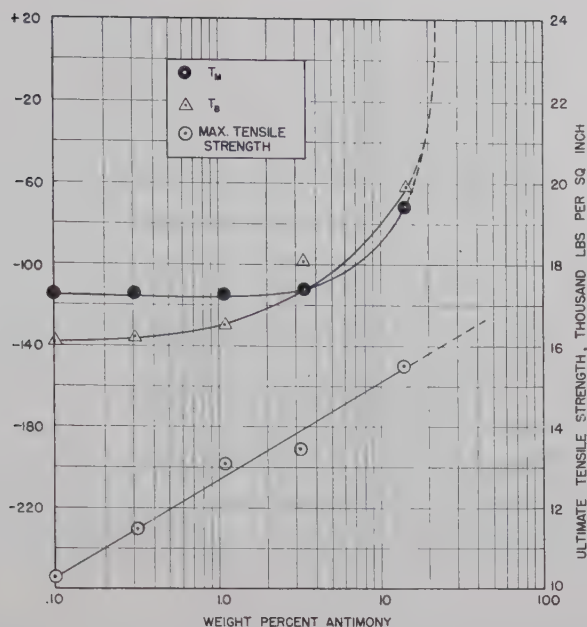


FIG 6—Variation of  $T_B$ ,  $T_M$ , and the maximum tensile strength at  $T_M$ , with composition.

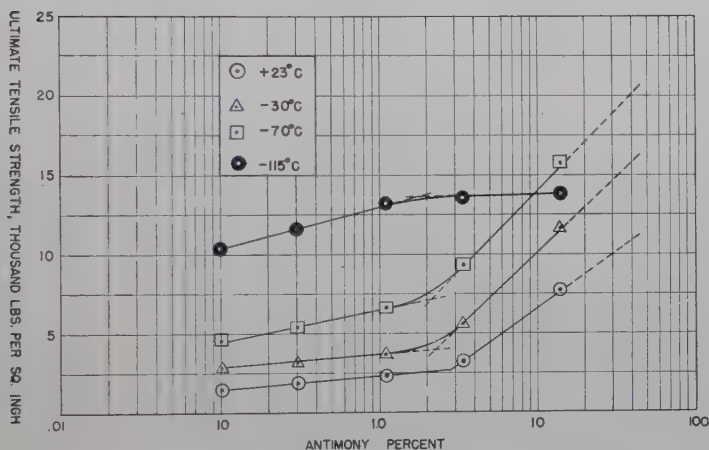


FIG 7—The effect at various temperatures of composition on the ultimate tensile strength of tin-antimony alloys containing from 0.1 to 10 pct antimony.

about the predominant  $\alpha$  matrix. The  $\alpha$  phase of the 20 pct alloy (Fig 1f), on the other hand, is even finer grained than that in the 10 pct alloy, and is composed of a network of both fine and agglomerated  $\gamma$  phase enclosing the white  $\alpha$  phase. In both these alloys, homogenization has caused extensive agglomeration, as indicated by the presence of large intergranular black particles along with the finer spheroidized grain-boundary precipitate also present. The eutectic alloy containing 33 pct cadmium, Fig 1g, is seen to contain even more of the black  $\gamma$  phase uniformly dispersed along with the finer  $\gamma$  spheroids in the  $\alpha$  matrix. Fig 1g shows the very interesting microstructure of the 60 pct cadmium

alloy. Here the large massive particles are primary  $\gamma$ , which appeared as massive dendrites in the cast structure. Homogenization has broken up these dendrites into very large particles of  $\gamma$ , although the distribution of the phase has been altered little from that in the cast structure. Also the lamellar structure lying between the  $\gamma$  dendrites in the cast alloy has been completely spheroidized and appears as a fine precipitate in the white  $\alpha$  matrix.

The influence of grain size on tensile properties has been investigated by other workers who used the total number of grains in the specimen cross-section as a variable.<sup>2</sup> For normal fine grain sizes, the effect is not significant, but tensile properties are considerably

influenced when the cross-section of the bar represents only a few grains. Since the base metal tin has a recrystallization temperature below room temperature for ordinary amounts of plastic strain, grain growth during the homogenizing anneal is possible. In the present case, however, both alloying elements raised the recrystallization temperature sufficiently so that no troublesome grain growth was encountered except in the 0.1 and 0.3 pct antimony alloys. Here the cross-section of the bar may frequently have contained only two or three grains, but in no case was a monocrystalline, chisel-type fracture obtained. The pronounced grain refining effect of cadmium on the cast structure resulted in extremely fine



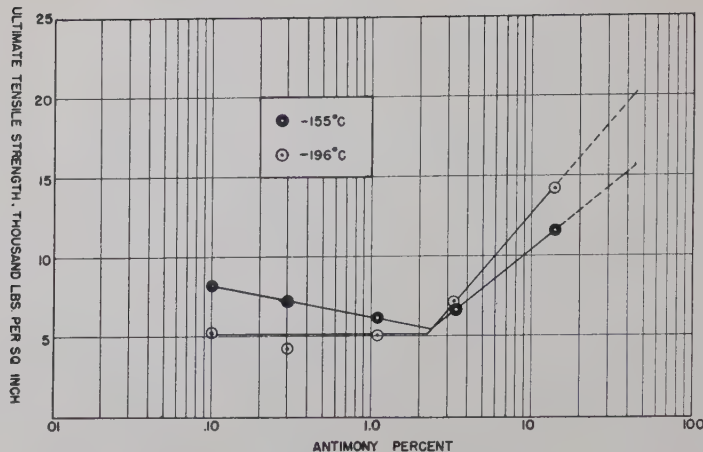


FIG 8—The effect at various temperatures of composition on the ultimate tensile strength of tin-antimony alloys containing from 0.1 to 10 pct antimony.

grain sizes in most of the cadmium alloys, especially those containing more than 3 pct cadmium.

### TESTING METHODS

Complete details of test specimen and testing procedure were those described in Ref. 1, except that isopentane was used for the immersion liquid for temperatures near  $-155^{\circ}\text{C}$ . The strain rate was maintained at 0.2 iipm for all tests, and testing temperatures were held to  $\pm 0.4^{\circ}\text{C}$ . The deviation of the measured load, using the weigh bar, was  $\pm 1.0$  lb or  $\pm 0.05$  pct at the highest load in all cases when the ductility was sufficient to allow ample time to balance the strain indicator. The elongation and reduction of area data represent the usual degree of accuracy attained in tensile testing, except that with coarse-grained fractures, the reduction of area is somewhat less accurate because of the difficulty in accurately measuring the final diameter.

As in the tin-lead studies<sup>1</sup> tensile specimens, 0.25 diam with 1 in. ga length, were tested either in duplicate or triplicate at each temperature and the results are summarized below in graphical form. In the event of considerable discrepancy between the duplicates, or if the fractured surface showed casting defects, a third test was made in nearly all cases.

### Discussion of Results

The data obtained in this investigation are presented graphically in Fig 3, 4, 5, 9, 10, and 11, the tensile strength

being plotted in various ways as functions of both temperature and composition. The pertinent data reported here include ultimate tensile strength, per cent elongation in a one inch gauge length, and per cent reduction in area. The values of true maximum stress were calculated for a few bars, but because of the experimental errors in the measurement of the true area and the doubtful accuracy of the breaking load values, no coherent data were obtained. Also, yield data are not reported because neither the "weigh bar," "drop-of-the-beam" or offset methods gave reproducible values.

In previous studies,<sup>1</sup> the low temperature tensile properties of pure tin were rationalized in terms of the effect of decreasing temperature on (1) the critical flow stress, (2) the extent or amount of cold work, and (3) the technical cohesive strength (true fracture stress at nil cold work). It is believed that a similar analysis can be made in the case of these data, with the stipulation, as given earlier, that when two or more phases are present in alloys, the measured tensile properties will be the net result of these three factors operating simultaneously on the separate phases. Thus the relative amount, form and distribution of the phases present will influence the magnitude of the net temperature effects. Also, since pure white tin is metastable below  $13.2^{\circ}\text{C}$ , all the tests except those at room temperature are subject to possible effects of the  $\alpha$  (white) to  $\beta$  (grey) transformation. It is known that rolling, bending or drawing enormously increases the velocity of transformation at  $-50^{\circ}\text{C}$ , the velocity being proportional to the intensity of deformation.<sup>11</sup>

In general, alloying elements are known to retard the transformation velocity but their effect on the equilibrium has never been determined. Both the kinetics and phase equilibria are being studied in this laboratory as major research problems; but until these data are available, it cannot be stated whether or not the present series of alloys were in a metastable condition when tested. However, in over 125 tests at sub-zero temperatures some of which lasted 15 min. or more, no visual evidence of the transformation from white to grey tin was observed, and it will therefore be eliminated as a variable.

As in the earlier work,<sup>1</sup> certain terms used in this discussion will be defined so that their use in this discussion will be clearly understood. Reduction in area, rather than elongation, will be used exclusively as the measure of ductility; temperature of embrittlement,  $T_B$ , is defined as the temperature at which 50 pct of maximum ductility, that is, 50 pct of maximum reduction in area, occurs in the given alloy. Temperature of maximum ultimate tensile strength,  $T_M$ , is self-defining. Technical cohesive strength is used here as the fracture stress at nil cold work. The critical flow stress is defined as the applied stress required to start flow.

To make this an integral report, a summary of the previous explanation of the pure tin properties will be given. For this purpose, Fig 2 containing the tensile properties of pure tin as a function of temperature from  $-195$  to  $+23^{\circ}\text{C}$  has been reproduced here. It has been postulated that for pure tin fracture at temperatures down to  $-125^{\circ}\text{C}$  occurred principally by flow, since 100 pct of the ductility remained

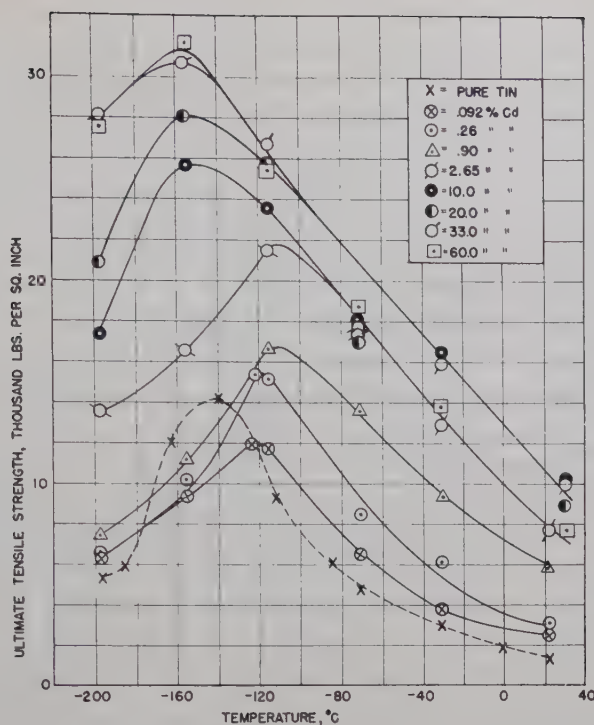


FIG 9—The effect of temperatures on the ultimate tensile strength of tin cadmium alloys containing from 0 to 60 pct cadmium.

at that temperature. Down to  $-60^{\circ}\text{C}$ , the rapid increase in tensile strength was caused partially from the increase of the flow stress with decreasing temperature, but principally from the increasing contribution of the work hardening, as evidenced by the increase in elongation to a maximum at this temperature. Below  $-60^{\circ}\text{C}$  the amount of work hardening decreased and the critical flow stress, increasing as the temperature decreased, became the main component of the tensile strength. At  $-130^{\circ}\text{C}$  as the ductility (R.A.) began falling off and approached 50 pct R.A., failure in tension occurred half by cleavage and half by flow, and the metal could then be considered brittle. At this point, work hardening was still contributing to the cleavage stress because considerable ductility still remained. However, as the ductility fell off, the contribution of the flow stress correspondingly decreased until failure occurred principally by cleavage. Finally, as the ductility decreased the cleavage stress simultaneously decreased and approached the true fracture stress at nil cold work.

An examination of the tensile data at the various subatmospheric temperatures for both the tin-antimony and tin-cadmium alloys indicates that the same three fundamental factors operating in the case of pure tin may also be responsible for the various alloy

properties. Of course, certain differences between the alloys and pure tin do exist, and these will be pointed out as the specific data for each system are considered. However, as in the case of the tin-lead alloys,<sup>1</sup> it will be assumed that because of the graphical analogies, the measured temperature effects on the properties of the alloys differ from those of pure tin only in degree. Hence the main emphasis in the following discussion will be placed on the correlation of the tensile data with composition and the explanation of these relations on the basis of microstructure.

The data for both binary systems have been presented graphically in various ways, as were the tin-lead data, in order to reveal pertinent relationships. These include (1) plots of tensile, elongation, and reduction in area or ductility values versus temperature, (2)  $T_B$ ,  $T_M$  vs. composition and maximum tensile strength vs. composition, the latter on a logarithmic scale, (3) tensile strength at each of the six test temperatures vs. log composition, and (4) for the tin-cadmium series only, log of tensile strength at each temperature level vs. composition on a linear scale.

For ease of reading, the same sequence as was followed in the earlier discussion will be adhered to here. That is, relationships between composition and tensile strength will be brought out by appropriate crossplots of the

data, and with the aid of these, the influence of composition on (1) the temperature of maximum tensile strength  $T_M$ , (2) the temperature of embrittlement  $T_B$ , (3) the maximum tensile strength at  $T_M$ , and (4) the tensile strength at the six testing temperatures will be described. A rationalization of these four specific composition-property relations in terms of the microstructures of the alloys will then be discussed in the indicated order.

#### TIN-ANTIMONY ALLOYS

It will be noted in Fig 3 that, for the tin-antimony alloys, increasing amounts of the second phase, that is, brittle  $\beta$  (SbSn) phase, has caused definite differences in the magnitude of the changes in tensile properties with decreasing temperature. On the other hand, the only significant difference between the shape of these curves and that for pure tin is that below  $-160^{\circ}\text{C}$ , where nil ductility is possessed by both pure tin or the tin-antimony alloys, the fracture stress increased with decreasing temperature for the 3 and 10 pct antimony alloys. This was also observed in the case of the 50-50 tin-lead alloys<sup>1</sup> as well as other metals, and further confirms the postulate that the fracture stress of metals in general increases with decreasing temperature.



Examination of the tensile data presented in Fig 3 shows that additions of 2.5 pct antimony and less had no measurable effect on the temperature of maximum tensile strength,  $T_M$ , or on the temperature of embrittlement,  $T_B$ . The higher antimony additions, however, definitely affected  $T_M$  and  $T_B$ , and these effects are obvious in Fig 6. Here it is seen that  $T_M$  and  $T_B$  are not synonymous in these alloys, the maximum difference being as much as 25°C for some alloys. As in the earlier work,<sup>1</sup> it is believed this difference is a result of the interaction of the three fundamental factors acting simultaneously either on the solid solutions of varying composition, or on the two phases present in some of the alloys.

In Fig 6 it can be seen that  $T_M$  remains essentially constant up through

3.38 pct antimony while  $T_B$  is consistently 25°C lower through 1 pct antimony. Then above 3.38 pct antimony for  $T_M$  and 1 pct for  $T_B$ , a rapid rise occurs, with  $T_B$  rising above  $T_M$  by from 10 to 15°C until at about 10 pct antimony they become synonymous. The maximum ultimate tensile strength (at  $T_M$ ) increases exponentially with increased antimony, as shown by the straight line relationship of the tensile strength at  $T_M$  vs. the logarithm of the composition (wt pct antimony). Variations of the tensile strength with composition at the six test temperatures is also of interest and is shown in Fig 7 and 8. Above  $T_B$ , additions of antimony up to about 2 pct had only a slight strengthening effect, but 3 pct antimony and higher caused an abrupt rise in the strength. These data for each

temperature seem to be represented satisfactorily by two straight lines intersecting between 2.2 and 2.8 pct antimony. At -115°C, additions of antimony up to about 1.8 pct have a strengthening effect but further additions had no apparent effect on the tensile strength. Below  $T_B$ , antimony contents up to 2 pct either decreased the strength slightly, as at -155°C, Fig 8, or have little or no effect, as at -196°C. Between 2.3 and 2.5 pct antimony, a sharp break in the curve is seen, with further additions of antimony causing a pronounced increase in strength.

These data and the various relationships between composition and tensile strength can be rationalized quite satisfactorily in terms of the heterogeneous microstructures developed in these alloys. Antimony additions up to about 2.5 pct did not affect  $T_M$  or  $T_B$  appreciably because the antimony remained in solid solution in tin. As mentioned earlier, the solid solubility of antimony in tin at room temperature has been reported recently as 3.5 pct by Hanson and Pell-Walpole.<sup>12</sup> However, the sharp break in the tensile strength vs. composition curves shown in Fig 7 and 8 may be interpreted as evidence that the solid solubility at 20°C lies between 2.2 and 2.8 pct antimony. The following explanation is thus offered: Antimony up to 2.2 pct had no effect on  $T_M$  or  $T_B$  because the alloys in this composition range were single phase. In this solid solution range, however, solution of antimony in the parent tin lattice formed a substitutional solid solution and strengthened the alloy in direct proportion to the percent antimony added at temperatures above  $T_B$ . Thus the maximum tensile strength and the ultimate tensile strength at and above -115°C increase with increased antimony. On the other hand, at -155°C and -196°C antimony in the solid solution range either decreases the tensile strength or has only a slight effect. Since both these temperatures are below  $T_B$ , the alloys in this composition range possess no measureable ductility and the observed tensile strength plotted in Fig 8 is really the true fracture stress of the alloys. These data thus indicate that antimony atoms, corresponding to 2 pct antimony or less, dissolved substitutionally in tin either slightly decrease or have little or no effect on their true fracture stress at temperatures below  $T_B$ .

Between 2.2 and 2.8 pct antimony, particles of  $\beta$  phase (SbSn) precipitated preferentially at the grain boundaries

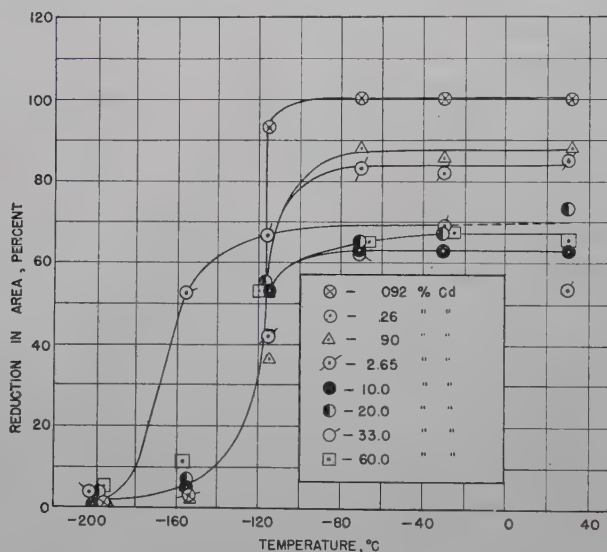


FIG 10—The effect of temperature on the reduction in area for tin-cadmium alloys.

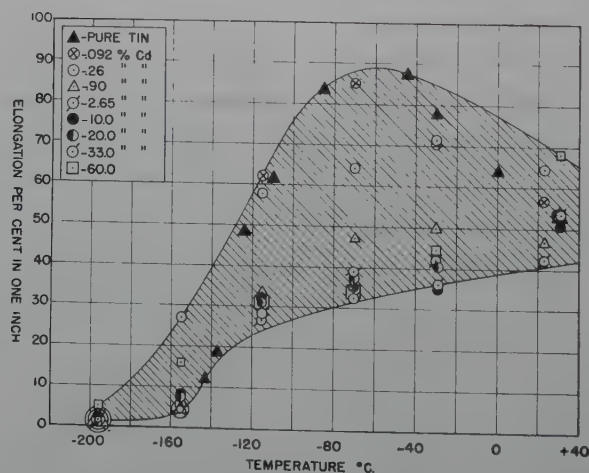


FIG 11—The percent elongation in one inch as a function of temperature for the cadmium alloys containing from 0 to 60 pct cadmium.

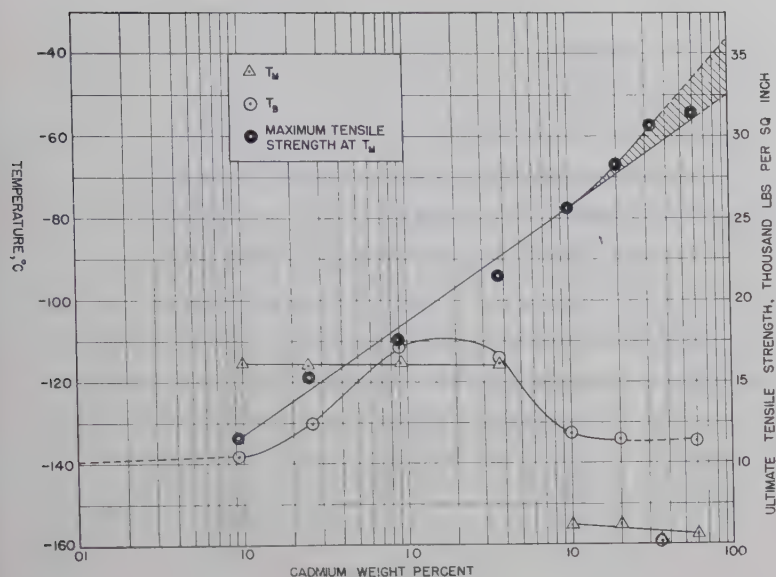


FIG 12—Variation of temperature of maximum tensile strength,  $T_M$ , temperature of embrittlement,  $T_B$ , and tensile strength at  $T_M$  with composition.

and this would be expected to cause precipitation hardening. The closest composition to this range included in this survey was a 3.38 pct alloy, and as seen in Fig 1b, this alloy was found to contain a grain boundary constituent, which was presumed to be particles of  $\beta$  phase. Property-wise, this was reflected by a slight rise in  $T_M$  and  $T_B$ , for here the  $\beta$  particles were either large or numerous enough to facilitate slip. However, the particles had not exceeded the critical particle size for hardening because the maximum tensile strength and the tensile strength above  $T_B$ , for this alloy, were higher than for the lower alloys. At the same time, at  $-115^\circ\text{C}$  the fact that the 3.38 pct alloy had no higher tensile strength than the 1.1 pct alloy at first appears anomalous. This is to be expected, however, since  $-115^\circ\text{C}$  is between  $T_B$  and  $T_M$ . The continuous  $\alpha$  (tin-rich) phase is of course predominant and is believed to determine the properties of this alloy. These results mean, then, that the true fracture stress of the  $\alpha$  tin-rich phase is the major remaining component of the tensile strength and in this case it is no higher than the combined critical flow stress, fracture stress and work hardening contribution making up the tensile strength of the lower solid solution alloys. Similarly the same explanation is offered to account for the ultimate tensile strength values obtained at  $-155$  and  $-196^\circ\text{C}$ .

In the 10.38 pct alloy, the  $\beta$  particles were considerably larger and more numerous, appearing intragranularly as

well as in the grain boundaries. These large brittle particles of  $\beta$  in the grain boundaries acted as discontinuities and consequently  $T_M$  and  $T_B$  increased abruptly. At the same time, sufficient intragranular precipitation occurred to cause the maximum tensile strength to increase. Above  $T_B$  the ultimate tensile strength also increased rapidly, presumably because as seen in Fig 4 and 5, considerable ductility remained so that critical flow stress and work hardening of both the continuous  $\alpha$  tin phase and discontinuous  $\beta$  phase was appreciably higher than that for the lower alloys. That the true fracture stress at this temperature remains small is indicated by the fact that at the lower tempera-

ture of  $-115^\circ\text{C}$ , that is,  $50^\circ\text{C}$  below  $T_B$  for this alloy, the net tensile strength, being essentially the true fracture stress (only 2 pct ductility remaining), is still equal only to the strength of the 3.38 pct alloy. Continuing this line of reasoning, at  $-155$  and  $-196^\circ\text{C}$ , the tensile strength increased appreciably because the fracture stress markedly increased with decreasing temperature. It is significant to note that the 10.38 pct antimony alloy was about 2500 psi stronger at  $-196^\circ\text{C}$  than at  $-155^\circ\text{C}$ , in further confirmation of the negative temperature coefficient of the true fracture stress.

Thus the variations with composition of  $T_M$ ,  $T_B$ , the maximum tensile strength at  $T_M$ , and the ultimate tensile strength at subatmospheric temperatures as low as  $-196^\circ\text{C}$  can be rationalized in a straightforward and logical manner in terms of the microstructure developed in the alloys. It is hoped that more fundamental data, such as true-stress strain measurements, yield data, tensile properties of variously strained alloys, creep data, and other information may soon be available to test the basic reasoning and perhaps separate the several variables used here necessarily in a qualitative way. Extension of this work to higher antimony alloys would be very desirable, especially from the fundamental standpoint. Extrapolation of the curves of Fig 6 indicates that  $T_M$  and  $T_B$  for the single phase  $\beta$  alloy (45 pct antimony) should be approximately  $+20^\circ\text{C}$ , and its maximum tensile strength should be about 16,500 psi. These predictions were confirmed in

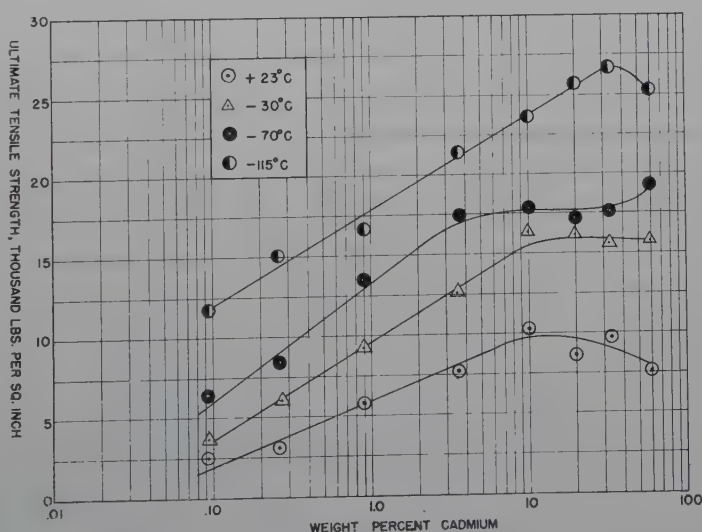


FIG 13—The effect at various temperatures of composition on the ultimate tensile strength of tin-cadmium alloys.



part when a 30 pct alloy was found to be brittle at about 0°C. However, since sound castings of this and higher compositions proved difficult to prepare, sufficient specimens were not available for these initial studies.

## TIN-CADMIUM ALLOYS

The tensile data for the tin-cadmium alloys containing from 0.01 to 60 pct cadmium are summarized graphically in Fig 9 to 11. Also, the curves for pure tin are superimposed on these for the tin-cadmium alloys for comparison purposes. As indicated above, a comparison of the tin-cadmium alloy curves with pure tin shows they are all similar in shape, except, of course, the magnitude of the effect of decreasing temperature on the tensile properties varies with the composition.

It will be noted first that the maximum in the tensile strength vs. temperature curve of Fig 9 for the alloys containing from 0.1 to 2.65 pct cadmium occurs at  $-115^{\circ}\text{C}$ , which is about  $25^{\circ}\text{C}$  higher than that for pure tin. Alloys containing from 10 to 60 pct cadmium appear to form another family of curves with the maximum at  $-155^{\circ}\text{C}$ . This apparent demarcation of the curves into two families with maxima from  $35$  to  $40^{\circ}\text{C}$  apart first appears to be anomalous, but it is believed to be caused by grain size differences rather than an effect of composition changes. The first four cadmium alloys were cast into slightly hotter molds than were the pure tin and the four higher cadmium alloys. Consequently, the grain size of the latter four alloys and tin was much finer and this not only shifted the maxima in tensile strength to a lower temperature, but raised the entire level of each tensile strength vs. temperature curve. Cursory microscopic examination confirmed this reasoning, but considerably more experimental work than was possible to include in this investigation would be required to establish more specifically the effect of grain size on low temperature tensile properties.

This effect must be considered to explain the data plotted in Fig 12 which shows the relation between  $T_M$ ,  $T_B$ , maximum tensile strength and composition. Two separate curves have been drawn for the  $T_M$  vs. composition data in line with the reasoning that the grain size of the four lower alloys was considerably larger than that of the four with higher cadmium contents. The effect of grain size, however, on the ductility and  $T_B$  of the eight alloys was

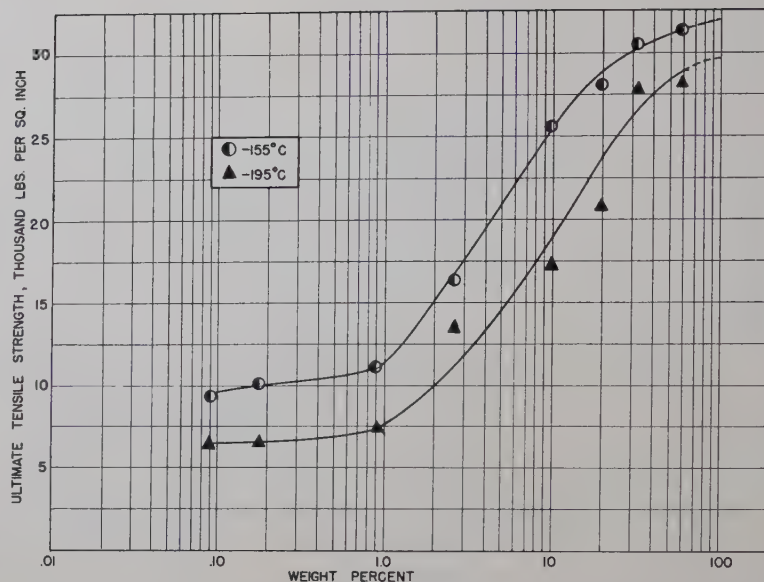


FIG 14—The effect of composition on the ultimate tensile strength of tin cadmium alloys at  $-155^{\circ}\text{C}$  and  $-195^{\circ}\text{C}$ .

not as pronounced as that on  $T_M$ . Composition had no apparent effect on  $T_M$  through 2.65 pct cadmium. Variation of the cadmium content from 10 to 60 pct, on the other hand, decreased  $T_M$  slightly.  $T_B$  follows a similar trend to that of the tin-lead<sup>1</sup> and tin-antimony alloys. Although the  $T_B$  vs. log of cadmium composition curve theoretically could only approach  $-140^{\circ}\text{C}$ , that is,  $T_B$  for pure tin at  $-\infty$  (since  $\log 0$  is  $-\infty$ ), an approximate extrapolation is pertinent to show that the first additions of cadmium up to 0.1 pct had little or no effect on  $T_B$ .  $T_B$  then increased steadily for the 0.26 and 0.90 pct alloys to a maximum between 1.0 and 2.65 pct cadmium followed by an abrupt decrease for the 10 to 60 pct alloys. A value for  $T_B$  constant within  $\pm 5^{\circ}\text{C}$  for pure tin was then approached. Finally an extremely low value for the eutectic 33 pct alloy was obtained. The maximum tensile strength at  $T_M$  increased uniformly over the entire composition range from 0.1 to 60 pct cadmium, the data following a straight line relationship quite well. The isothermal tensile strength-composition data plotted in Fig 13 and 14 show that above  $T_B$ , the tensile strength increases as the cadmium content increases, the curve approaching maxima at about 10 pct cadmium and either decreasing slightly or remaining constant for the 20, 33 and 60 pct cadmium compositions. At  $-115^{\circ}\text{C}$ , pronounced strengthening from cadmium additions continues and obeys a straight line relationship until a maximum is reached at 33 pct fol-

lowed by a decrease in strength for the 60 pct alloy. Below  $T_B$ , that is, at  $-155$  and  $-196^{\circ}\text{C}$ , however, additions of cadmium up to 1 pct have only a slight strengthening effect. Then a sharp break occurs at the 1 pct composition level, with further additions causing very marked increases in tensile strength to either approach or attain maxima at from 33 to 60 pct cadmium. As in the case of the tin-lead<sup>1</sup> and tin-antimony systems, this break in the tensile strength vs. log composition below  $T_B$  corresponds with the terminal solid solution limit of cadmium in tin, and is taken as confirmation of this 10 pct solid solubility of cadmium in tin reported by Hanson and Pell-Walpole.<sup>5</sup> Further, if the tensile data for temperatures above  $T_B$  are replotted with the tensile strength on a logarithmic scale, a very definite break in the curves occurs at about 1 pct cadmium, Fig 15. These data are also considered to confirm the 1 pct cadmium solid solubility in tin. It is undoubtedly significant that two distinctly different relationships between tensile strength and composition are obeyed for above and below  $T_B$  respectively, but without additional fundamental data it can only be speculated that this difference is related to the fact that the fundamental process operating above  $T_B$  is cleavage by flow, whereas below  $T_B$ , failure occurs principally by fracture. At any rate, having confirmed the most recent tin-cadmium phase diagram reported in the literature,<sup>5</sup> the following correlation of low temperature tensile properties and

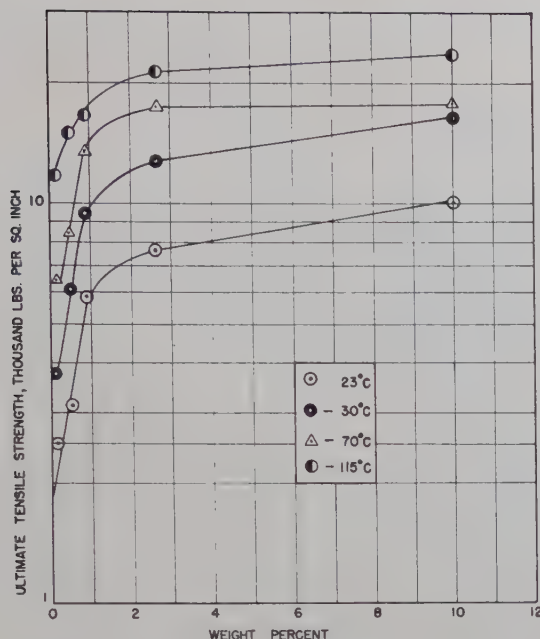


FIG 15—The ultimate tensile strength for various temperatures as a function of composition in the range from 0 to 10 pct cadmium.

microstructure is offered. All the data and relationship revealed by various graphical treatments will be considered except  $T_M$  vs. composition curve since, because of grain size effects, it is somewhat anomalous.

The first additions of cadmium (up to 0.1 pct) had no effect on  $T_B$ , however, because the substitutional cadmium atoms were too few to change the flow-fracture properties of the resulting solid solution from those of the pure tin. Increasing numbers of cadmium atoms, however, corresponding to 0.26 and 0.9 pct cadmium distorted the parent tin lattice increasingly, so that cleavage by flow was facilitated and  $T_B$  increased. The tensile strength at  $T_M$  also increased continuously in this composition range, presumably because increased numbers of cadmium atoms dissolved substitutionally increased the critical flow stress and work-hardening contribution of these homogeneous solid solutions. The tensile strengths of these alloys above  $T_B$  also increased progressively for the same reason. Below  $T_B$ , however, increasing cadmium up to 0.9 pct had only a slight strengthening effect, indicating that dilution of the tin-rich matrix with substitutional cadmium atoms did not appreciably influence the true fracture stress values of the various alloys.

At 1.0 pct cadmium, however, a fine precipitate of  $\gamma$  phase began appearing preferentially at the grain boundaries, slip was thus facilitated and  $T_B$  in-

creased to a maximum between 1 and 3 pct cadmium. The maximum tensile strength continued to increase, however, probably because of precipitation hardening and because the fracture stress of the  $\gamma$  phase, occurring both inter- and intragranularly in the 2.65 pct alloy, was sufficient to supplement the composite flow, work hardening, and fracture stress of the continuous tin phase. The tensile strength above  $T_B$  continued to increase in the range from 0.1 to 3 pct cadmium, probably because of precipitation hardening. At  $-155$  and  $-196^\circ\text{C}$ , however, or below  $T_B$ , the increase in tensile strength must be attributed to the higher combined fracture strengths of the precipitated  $\gamma$  and the continuous  $\alpha$  phases. It is significant to note that as  $T_B$  is more nearly approached or exceeded, the slopes of the curves of Fig 13 increase in the 0.1 to 3 pct cadmium range. This can be interpreted as indicating that the technical cohesive strength component of the net tensile strength is increasing and becoming more predominant with decreasing temperature. Ultimately, the tensile strengths at  $-155$  and  $-196^\circ\text{C}$  are the true fracture strengths, since no ductility remains at these low temperatures.

As the composition reached and exceeded 2.65 pct cadmium, the intergranular  $\gamma$  precipitate increased in amount so that flow was facilitated in this phase as well as the  $\alpha$  phase and thence  $T_B$  decreased to a constant

value of about  $-134^\circ\text{C}$  at the 10 pct cadmium level. The maximum tensile strength increased in the 2.65 to 10 pct cadmium range probably because the  $\gamma$  phase was uniformly distributed and the critical flow stress and work-hardening of the two-phase alloy increased with increasing amounts of  $\gamma$  phase. The tensile strengths at  $+23$  and  $-30$  were also improved because both the  $\alpha$  and  $\beta$  phases had higher critical flow stresses and were strengthened by work hardening to give higher tensile strengths with increasing cadmium contents. At  $-70^\circ\text{C}$ , however, the  $\gamma$  phase, appearing in increasing amounts in the 2.65 to 10 pct range to form a grain boundary envelope, was probably brittle and the tensile strength of the alloy was thus determined by the strength of the continuous tin phase. Since the composition of this  $\alpha$  tin phase remains essentially constant, its strength remained constant as shown by the fact that the tensile strength of the nominal 2.65 and 10 pct cadmium alloys is essentially constant. At  $-115^\circ\text{C}$ , the fracture stress of the intergranular  $\gamma$  phase increased, so that the net tensile strength of the 10 pct alloys is higher, and this strengthening, caused by the increase of the fracture stress of both the  $\alpha$  and  $\gamma$  phases, increased in degree as the temperature decreased. This accounts for the increasing slopes of the tensile strength vs. log composition curves as the temperature decreased.

In the 20 and 33 pct alloys, the  $\gamma$  phase was agglomerated into quite massive particles which probably became brittle before the continuous  $\alpha$  phase, as suggested above. Thus since the composition of the  $\alpha$  phase remained constant in the 10–20 and 33 pct alloys  $T_B$  also remained constant. The maximum tensile strength, however, did continue to increase, presumably because the fracture stress of the increasing  $\gamma$  phase contributed appreciably, that is, corresponding to the amount of  $\gamma$  present. The tensile strength at  $+23$ ,  $-30$  and  $-70$  remains essentially constant in the 10 to 33 pct composition range, either (1) because the massive  $\gamma$  phase is brittle and its fracture stress at all these temperatures is low so that the tensile strength of the alloy was determined by the constant composition  $\alpha$  phase, or (2) at  $+23$  and  $-30$ , the critical flow stress, work hardening contribution and the fracture stress of the  $\gamma$  phase is less than those of the  $\alpha$  phase, and the latter still determined the tensile strength of the alloy. At



−115°C, the fracture stress of the  $\gamma$  phase increased and the net tensile strength thus increased. At −155 and −196°C, that is, below the embrittlement temperature for both the  $\alpha$  and  $\gamma$  phases, the increase of fracture stress with decreasing temperature accounts satisfactorily for the increase in tensile strength in the 10 to 33 pct cadmium range.

Again, in the case of the 60 pct cadmium alloy, the continuous  $\alpha$  phase determined  $T_B$ , and thus, since the tin matrix is of constant composition, as in the 10 pct higher alloy,  $T_B$  remained constant. The maximum tensile strength continued to increase, however, for the same reasons as given for those in the 10 to 33 pct range. The tensile strengths at +23 and −30°C decrease slightly for the 60 pct alloy, probably because the critical flow stress, work-hardening contribution and fracture stress of the massive  $\gamma$  phase is considerably less than the corresponding factors for the  $\alpha$  phase. At −70°C, the 60 pct alloy is slightly stronger as a result of the increase of the fracture stress of the  $\gamma$  phase with decreasing temperature. On the other hand, at −115°C the tensile strength decreased for this 60 pct alloy, possibly because the decrease of the work hardening component with decreasing temperature was greater than the increase of fracture strength accompanying this fall in temperature. The decrease of strength for this alloy at −155°C cannot be explained according to this reasoning, and thence must be considered anomalous until more information is available. At −196°C, the abrupt improvement in strength for this alloy is undoubtedly a result of the increase of the fracture strength of both phases with decreased temperature.

This correlation of microstructure and low temperature tensile properties has necessarily been qualitative, but it is hoped that these rationalizations may be helpful to design engineers who need to know the properties of similar white bearing alloys for low temperature service. Certain anomalies have been pointed out throughout the discussion, and as might be expected in such survey investigations, many other undelineated questions must go unanswered at this time. A rather extensive long range fundamental research program on low temperature phenomena is underway in this laboratory, however, and it is hoped that a rigorous analysis of these and other related low

temperature behaviors will soon be forthcoming.

## Conclusions

1. The tin-antimony alloys containing up to 3 pct antimony remain ductile down to −100°C, whereas the 10 pct antimony alloy becomes brittle at −60°C. Extrapolation would indicate that pure  $\beta$ (SbSn) is brittle at room temperature, a fact confirmed by other workers.<sup>3</sup> Tin-cadmium alloys containing from 0.01 to 2.65 pct cadmium become brittle below −120°C, while the 10 to 60 pct cadmium alloys as tested did not become brittle until about −155°C was reached. Since these higher cadmium alloys were finer grained than the lower alloys, this behavior was interpreted as a grain size effect superimposed on a composition effect.

2. All the tin-binary alloys tested increased in strength as the temperature decreased prior to embrittlement. This increase in tensile strength with decreasing temperature has been explained on the basis of (1) the increase of the critical flow stress with decreasing temperature, (2) the effects of work hardening accompanying plastic deformation below the recrystallization temperature, and (3) the increase of technical cohesive strength (fracture stress at nil cold work) with decreasing temperature. The properties measured, of course, were the net or composite strengths arising from the operation of these three factors on the two or more phases present in most of the alloys.

3. Antimony in solid solution has little effect on the tensile strength at any temperature above and below the embrittlement temperature, but as a second phase, it greatly strengthens the alloys. Cadmium, on the other hand, has an appreciable strengthening effect above the embrittlement temperature, whether in solid solution or as a second phase. Below the embrittlement temperature, however, solid-solution strengthening is only slight, whereas cadmium-rich second phase ( $\gamma$ ) increases the strength markedly.

4. Antimony raises the embrittlement temperature as soon as an antimony-rich phase appears. Cadmium, either in solid solution or as a second phase, has little effect on the temperature of embrittlement.

5. The use of low temperature tensile tests to verify the solvus lines of

binary phase diagrams near room temperature has been demonstrated both in the tin-antimony and tin-cadmium systems.

## Acknowledgment

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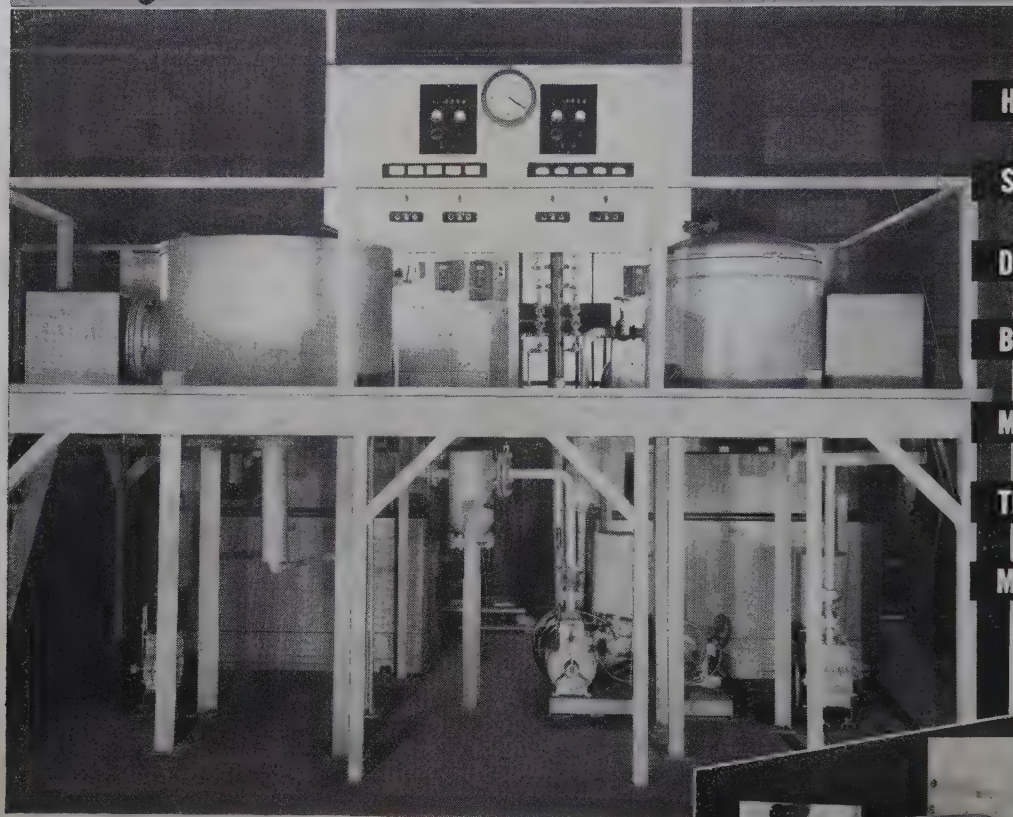
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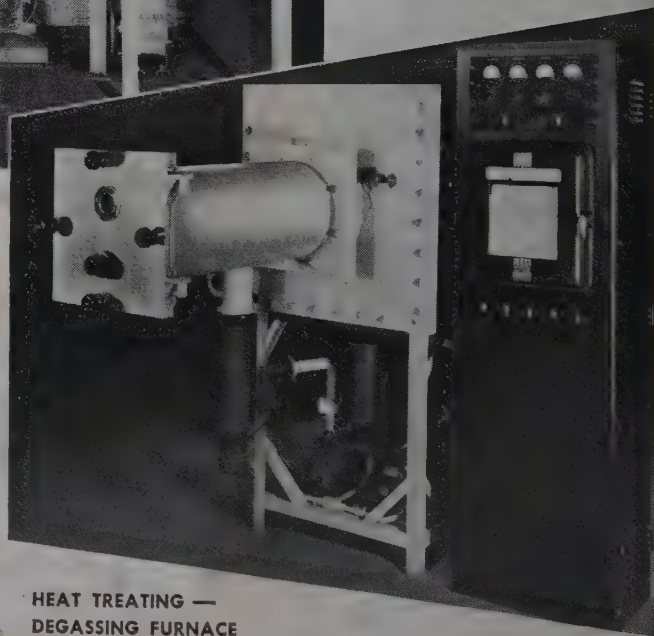
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